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NEWS	2	AUG 10	Time limit for inactive STN sessions doubles to 40 minutes
NEWS	3	AUG 18	COMPENDEX indexing changed for the Corporate Source (CS) field
NEWS	4	AUG 24	ENCOMPLIT/ENCOMPLIT2 reloaded and enhanced
NEWS	5	AUG 24	CA/Caplus enhanced with legal status information for U.S. patents
NEWS	6	SEP 09	50 Millionth Unique Chemical Substance Recorded in CAS REGISTRY
NEWS	7	SEP 11	WPIDS, WPINDEX, and WPIX now include Japanese FTERM thesaurus
NEWS	8	OCT 21	Derwent World Patents Index Coverage of Indian and Taiwanese Content Expanded
NEWS	9	OCT 21	Derwent World Patents Index enhanced with human translated claims for Chinese Applications and Utility Models
NEWS	10	NOV 23	Addition of SCAN format to selected STN databases
NEWS	11	NOV 23	Annual Reload of IFI Databases
NEWS	12	DEC 01	FRFULL Content and Search Enhancements
NEWS	13	DEC 01	DGENE, USGENE, and PCTGEN: new percent identity feature for sorting BLAST answer sets
NEWS	14	DEC 02	Derwent World Patent Index: Japanese FI-TERM thesaurus added
NEWS	15	DEC 02	PCTGEN enhanced with patent family and legal status display data from INPADOCDB
NEWS	16	DEC 02	USGENE: Enhanced coverage of bibliographic and sequence information
NEWS	17	DEC 21	New Indicator Identifies Multiple Basic Patent Records Containing Equivalent Chemical Indexing in CA/Caplus
NEWS	18	JAN 12	Match STN Content and Features to Your Information Needs, Quickly and Conveniently
NEWS	19	JAN 25	Annual Reload of MEDLINE database
NEWS	20	FEB 16	STN Express Maintenance Release, Version 8.4.2, Is Now Available for Download
NEWS	21	FEB 16	Derwent World Patents Index (DWPI) Revises Indexing of Author Abstracts
NEWS	22	FEB 16	New FASTA Display Formats Added to USGENE and PCTGEN
NEWS	23	FEB 16	INPADOCDB and INPAFAMDB Enriched with New Content and Features
NEWS	24	FEB 16	INSPEC Adding Its Own IPC codes and Author's E-mail Addresses

10/591,778

NEWS EXPRESS FEBRUARY 15 10 CURRENT WINDOWS VERSION IS V8.4.2,  
AND CURRENT DISCOVER FILE IS DATED 15 JANUARY 2010.

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\* \* \* \* \* STN Columbus \* \* \* \* \*

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ENTRY	SESSION
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FILE 'CAPLUS' ENTERED AT 08:24:45 ON 27 FEB 2010

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FILE COVERS 1907 - 27 Feb 2010 VOL 152 ISS 10

FILE LAST UPDATED: 26 Feb 2010 (20100226/ED)

REVISED CLASS FIELDS (/NCL) LAST RELOADED: Dec 2009

USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Dec 2009

CAPlus now includes complete International Patent Classification (IPC)  
reclassification data for the third quarter of 2009.

CAS Information Use Policies apply and are available at:

<http://www.cas.org/legal/infopolicy.html>

This file contains CAS Registry Numbers for easy and accurate

10/591,778

substance identification.

```
=> s sterically(1)hindered(1)aminoxide
    19192 STERICALLY
    44142 HINDERED
```

```
    93 AMINOXIDE
L1      2 STERICALLY(L)HINDERED(L)AMINOXIDE
```

```
=> s sterically(1)hindered(1)amine(1)ether
    19192 STERICALLY
    44142 HINDERED
    316044 AMINE
    575130 ETHER
```

```
L2      47 STERICALLY(L)HINDERED(L)AMINE(L)ETHER
```

```
=> s 1 or 2
    10554952 1
    10425547 2
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SYSTEM LIMITS EXCEEDED - SEARCH ENDED

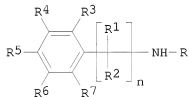
The search profile you entered was too complex or gave too many answers. Simplify or subdivide the query and try again. If you have exceeded the answer limit, enter DELETE HISTORY at an arrow prompt (=>) to remove all previous answers sets and begin at L1. Use the SAVE command to store any important profiles or answer sets before using DELETE HISTORY.

```
=> s l1 or l2
L3      48 L1 OR L2
```

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=> d l3 1-48 bib abs
```

L3 ANSWER 1 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2010:147002 CAPLUS  
 TI Absorbent solution based on a tertiary or hindered amine and on one particular activator and process for removing acid compounds from a gaseous effluent  
 IN Jacquin, Marc; Grandjean, Julien; Huard, Thierry  
 PA IFP, Fr.  
 SO PCT Int. Appl., 32pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA French  
 FAN.CNT 4

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2010012884	A1	20100204	WO 2009-FR902	20090721
	W: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW				
	RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LI, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, SE, SI, SK, SM, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	FR 2934172	A1	20100129	FR 2008-4304	20080728
PRAI	FR 2008-4304	A	20080728		
GI					



AB The invention relates to the removal of acid compds. in a gaseous effluent in an absorption process that uses an aqueous solution comprising a tertiary amine or a sterically hindered amine as a mixture with a primary or secondary amine corresponding to the general formula (I), where  $n = 1$  or  $2$ , and each of the groups  $R_1$ ,  $R_2$ ,  $R_3$ ,  $R_4$ ,  $R_5$ ,  $R_6$ ,  $R_7$  and  $R$  is chosen independently from one of the group consisting of: a hydrogen atom, a linear, branched or cyclic C1-12 alkyl group, an aryl group, a hydroxyalkyl group or a linear, branched or cyclic C1-12 ether oxide group. The invention advantageously applies for the treatment of natural gas and of gas of industrial origin.

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 2 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2008:1529938 CAPLUS  
 DN 150:80828  
 TI Nitroxide containing electrode materials for secondary batteries  
 IN Nesvadba, Peter; Bugnon Folger, Lucienne; Hintermann, Tobias  
 PA Ciba Holding Inc., Switz.  
 SO PCT Int. Appl., 58pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2008155247	A1	20081224	WO 2008-EP57138	20080609
W: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW, RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, IG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
PRAI EP 2007-110574	A	20070619		
OS MARPAT 150:80828				
AB This invention relates to a stable secondary battery utilizing as active principle the oxidation and reduction cycle of a sterically hindered nitroxide radical, a sterically hindered oxoammonium cation, a sterically hindered hydroxylamine or a sterically hindered aminoxide anion containing a piperazin-2,6-dione, a piperazin-2-one or morpholin-2-one structural unit. Further aspects of the invention are a method for providing such a secondary battery, the use of the resp. compds. as active elements in secondary batteries and selected novel compds.				
RE.CNT 10	THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT			

L3 ANSWER 3 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2008:941646 CAPLUS

DN 149:227491

TI Desulfurization of natural gas by absorption

IN Magne-Drisch, Julia; Lucquin, Anne-Claire; Streicher, Christina; Elgue, Jean; Cousin, Jean-Paul; Perdu, Gauthier; Roquet, Damien; Dinh, Viep Hoang

PA Institut Francais du Pretrrole, Fr.; Total SA

SO U.S. Pat. Appl. Publ., 17 pp.

CODEN: USXXCO

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----		-----	-----	-----
PI	US 20080187485	A1	20080807	US 2007-671650	20070206
PRAI	US 2007-671650		20070206		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB Hydrogen sulfide contained in a hydrocarbon gas, especially natural gas, containing

traces of aromatic hydrocarbons is removed by (a) contacting the gas with an absorbent solution to obtain a gas depleted in H<sub>2</sub>S and a H<sub>2</sub>S-rich absorbent solution, (b) heating and expanding the H<sub>2</sub>S-rich absorbent solution to a predetd. temperature and pressure to release a gaseous fraction containing

aromatic

hydrocarbons and to obtain an absorbent solution depleted in aromatic hydrocarbons, with the temperature and pressure being selected such that the gaseous fraction contains at least 50% of the aromatic hydrocarbons and at most 35% hydrogen sulfide contained in the H<sub>2</sub>S-rich absorbent solution, (c) thermally regenerating the absorbent solution depleted in aromatic hydrocarbon compds. to release a H<sub>2</sub>S-rich gaseous effluent and to obtain a regenerated absorbent solution At least a part of the H<sub>2</sub>S-rich gaseous effluent of stage (c) is treated by a Claus process. The absorbent is an aqueous amine solution The amine can be methyldiethanolamine, diisopropanolamine, monoethanolamine, diethanolamine, or a sterically hindered amine at a temperature of 80-140° and a pressure of 1.5-6 bara. The absorbent can be sulfolane, methanol, N-formylmorpholine, acetylmorpholine, propylene carbonate, polyethylene glycol di-Me ether, or N-Me pyrrolidone.

L3 ANSWER 4 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2008:43070 CAPLUS  
 DN 148:145644  
 TI Process for the preparation of sterically hindered nitroxyl ethers  
 IN Basbas, Abdel-Ilah; Alvisi, Davide; Cordova, Robert; DiFazio, Michael  
 Peter; Fischer, Walter; Kotrola, Joseph A.; Nocentini, Tiziano; Robbins,  
 James; Schoening, Kai-Uwe  
 PA Ciba Specialty Chemicals Holding Inc., Switz.  
 SO PCT Int. Appl., 38pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2008003605	A1	20080110	WO 2007-EP56301	20070625
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW				
	RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	CA 2654837	A1	20080110	CA 2007-2654837	20070625
	EP 2035382	A1	20090318	EP 2007-786825	20070625
	EP 2035382	B1	20091118		
	R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR, AL, BA, HR, MK, RS				
	JP 2009541429	T	20091126	JP 2009-517150	20070625
	AT 449072	T	20091215	AT 2007-786825	20070625
	KR 2009031523	A	20090326	KR 2008-731543	20081226
	CN 101484423	A	20090715	CN 2007-80025374	20090104
	IN 2009CN00058	A	20090605	IN 2009-CN58	20090105
	MX 2009000047	A	20090123	MX 2009-47	20090107
PRAI	EP 2006-116619	A	20060705		
	EP 2007-106899	A	20070425		
	WO 2007-EP56301	W	20070625		
OS	CASREACT 148:145644; MARPAT 148:145644				
GI					

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB The present invention relates to a novel process for the preparation of specific sterically hindered nitroxyl ethers from their corresponding sterically hindered nitroxyl radicals by reacting it with an aldehyde and a hydroperoxide. This nitroxyl ether formation may be carried out from different starting nitroxyl radicals, which are subsequently further reacted to the desired compds. The compds. prepared by this process are effective as stabilizers for polymers against harmful effects of light, oxygen and/or heat and as flame-retardants for polymers. Thus, a process for the preparation

of a sterically hindered nitroxyl ether of formula I, wherein n is a number from 1 to 10 and R1 is C1-C5 alkyl, comprises the steps of (a) reacting the nitroxyl compound with n-hexylaldehyde, n-pentylaldehyde, n-butyraldehyde, n-propylaldehyde or acetaldehyde and a hydroperoxide in the presence of a metal catalyst; or (b) reacting 1-oxyl-2,2,6,6-tetramethylpiperidin-4-one with n-hexylaldehyde, n-pentylaldehyde, n-butyraldehyde, n-propylaldehyde or acetaldehyde or a mixture of said aldehydes with their resp. alcs. and a hydroperoxide in the presence of a metal catalyst to yield 1-alkoxy-2,2,6,6-tetramethylpiperidin-4-one, which is further reacted to form a sterically hindered nitroxyl ether of formula I. Thus, a process for the preparation of a sterically hindered nitroxyl ether of formula II, wherein N4-amine is  $H_2N-(CH_2)_3-NH-(CH_2)_2-NH-(CH_2)_3-NH_2$ , is by reacting a compound of formula 1-oxyl-2,2,6,6-tetramethylpiperidin-4-ol or 1-oxyl-2,2,6,6-tetramethylpiperidin-4-one with a compound of formula 1,2,3,6-Tetrahydrobenzaldehyde or cyclohexanecarboxaldehyde and a hydroperoxide in the presence of a metal catalyst to yield the 1-cyclohex-3-enyloxy/1-cyclohexyloxy of 2,2,6,6-tetramethylpiperidin-4-one/ol, which are further reacted to form a compound of formula II.



L3 ANSWER 5 OF 48 CAPLUS COPYRIGHT 2010 ACS ON STN  
 AN 2007:435706 CAPLUS  
 DN 146:422837  
 TI Production of sterically hindered amine ethers useful as stabilizers for  
 polymer compositions  
 IN Frey, Markus; Rast, Valerie  
 PA Ciba Specialty Chemicals Holding Inc., Switz.  
 SO PCT Int. Appl., 89pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2007042422	A2	20070419	WO 2006-EP66962	20061002
	WO 2007042422	A3	20070607		
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW			
	RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AP, EA, EP, OA			
	EP 1943236	A2	20080716	EP 2006-806923	20061002
	R:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR			
	JP 2009514803	T	20090409	JP 2008-534986	20061002
	US 20090318592	A1	20091224	US 2009-83052	20090304
PRAI	EP 2005-109432	A	20051011		
	WO 2006-EP66962	W	20061002		

## ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

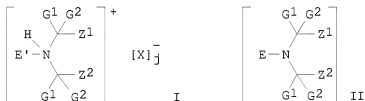
OS MARPAT 146:422837

AB The invention relates to processes for preparation of sterically hindered amine ethers by reacting a sterically hindered amine oxide with a ketone or an aldehyde having at least one reactive H in the presence of a peroxydisulfate. The products obtained may be hydrogenated. The compds. made by these processes are particularly effective in stabilization of polymer compns. against harmful effects of light, oxygen, heat, and as flame-retardants for polymers. Thus, 1-[(2,2,6,6-tetramethyl-1-piperidinyl)oxy]-2-propanone was prepared in 82% (66% isolated) yield by adding TEMPO (7.8) and acetone (30.5) to a stirred solution of silver nitrate (0.17 g) in deionized water (30 mL) at 25°, bringing the mixture to reflux (62°), slowly adding a solution of sodium peroxydisulfate (11.9 g) in water (45 mL) over 3 h, cooling the mixture to 25°, and neutralizing with sodium bicarbonate.

L3 ANSWER 6 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2006:823408 CAPLUS  
 DN 145:250371  
 TI Stabilization of natural composites containing cellulosic or wood based fillers with sterically hindered amine  
 IN Kaspers, Sarah R.; Guckel, Christian; Rogez, Daniel; Schaller, Christian M.  
 PA Ciba Specialty Chemicals Corp., USA  
 SO U.S. Pat. Appl. Publ., 37 pp.  
 CODEN: USXXCO  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 20060183821	A1	20060817	US 2006-352873	20060213
	WO 2006087269	A1	20060824	WO 2006-EP50673	20060206
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
	RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
	DE 112006000348	T5	20071227	DE 2006-112006000348	20060206
	JP 2008531324	T	20080814	JP 2007-555572	20060206
PRAI	US 2005-653170P	P	20050215		
	WO 2006-EP50673	W	20060206		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT  
 OS MARPAT 145:250371  
 GI



AB A method of protecting natural composites against light-induced degradation comprises the steps of: treating cellulosic or wood based fillers with an impregnating composition wherein the impregnating composition comprises (a) water or

water/organic solvent and (b) a sterically hindered amine which is selected from at least one of the compds. of formulas I and II, wherein G<sup>1</sup> and G<sup>2</sup> are independently alkyl of 1 to 4 carbon atoms or are together pentamethylene; Z<sup>1</sup> and Z<sup>2</sup> are each Me, or Z<sup>1</sup> and Z<sup>2</sup> together form an unsubstituted linking moiety or a linking moiety substituted by one or more groups selected from an ester, ether,

hydroxy, oxo, cyanohydrin, amide, amino, carboxy or an urethane group; E is oxyl, hydroxyl, hydrogen or methyl; E' is hydroxyl, hydrogen or methyl; X is an inorg. or organic acid; and, wherein the total charge of h cations is equal to the total charge of j anions; drying the impregnated fillers, and blending the impregnated fillers with a polymer via compounding methods such as extrusion to form the natural composite.

L3 ANSWER 7 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2006:440107 CAPLUS  
 DN 144:468916  
 TI Process for the synthesis of sterically hindered N-alkoxyamines  
 IN Frey, Markus; Rast, Valerie; Martinez, Francisco; Alvisi, Davide  
 PA Ciba Specialty Chemicals Holding Inc., Switz.  
 SO PCT Int. Appl., 75 pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2006048389	A1	20060511	WO 2005-EP55472	20051024
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
EP 1807395	A1	20070718	EP 2005-801650	20051024
R:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR			
CN 101048378	A	20071003	CN 2005-80037093	20051024
JP 2008519003	T	20080605	JP 2007-539568	20051024
US 20090069470	A1	20090312	US 2007-665885	20070419
PRAI EP 2004-105456	A	20041102		
WO 2005-EP55472	W	20051024		

## ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS MARPAT 144:468916

AB The present invention relates to novel processes for the preparation of a sterically hindered amine ethers by the transformation of a corresponding oxo-piperidin to a hydroxy or amino substituted sterically hindered amine ether and the preparation of a N-propoxy or N-propenoxy substituted sterically hindered amine and some novel compds. obtainable by these processes. The compds. made by these processes are particularly effective in the stabilization of polymer compns. against harmful effects of light, oxidation and/or heat and as flame-retardants for polymers.

OSC.G 3 THERE ARE 3 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 8 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2005:1042220 CAPLUS  
 DN 143:347055  
 TI A process for the synthesis of sterically hindered amine ethers useful as stabilizing and fireproofing agents  
 IN Frey, Markus; Rast, Valerie; Braig, Adalbert; Kramer, Andreas  
 PA Ciba Specialty Chemicals Holding Inc., Switz.  
 SO PCT Int. Appl., '71 pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2005090307	A1	20050929	WO 2005-EP50995	20050307
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
	RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
	DE 112005000536	T5	20070201	DE 2005-112005000536	20050307
	JP 2007529467	T	20071025	JP 2007-503327	20050307
	US 20070191516	A1	20070816	US 2006-591778	20060906
PRAI	EP 2004-101047	A	20040315		
	WO 2005-EP50995	W	20050307		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS CASREACT 143:347055; MARPAT 143:347055

AB A process for the preparation of a sterically hindered amine ether which comprises reacting a corresponding sterically hindered aminoxide with a C5-C18alk-1-ene in the presence of an organic hydroperoxide and optionally hydrogenating the resulting product as well as the product mixts. obtained therewith and their use as stabilizers and flame retardants.

OSC.G 2 THERE ARE 2 CAPLUS RECORDS THAT CITE THIS RECORD (5 CITINGS)

RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 9 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2005:601518 CAPLUS

DN 143:99285

TI Sulphur scavenging amines being monomeric adducts of a sterically hindered amine and an aldehyde or donor thereof

IN Gatlin, Larry W.

PA Clearwater International LLC., USA

SO Brit. UK Pat. Appl., 39 pp.

CODEN: BAXXDU

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	GB 2409859	A	20050713	GB 2005-156	20050106
	GB 2409859	B	20090401		
	US 20050153846	A1	20050714	US 2004-754487	20040109
	US 7517447	B2	20090414		
	CA 2491973	A1	20050709	CA 2005-2491973	20050107
	NO 2005000101	A	20050711	NO 2005-101	20050107
	AU 2005200048	A1	20050728	AU 2005-200048	20050107
	US 20090250659	A	20091008	US 2009-419418	20090407
PRAI	US 2004-754487	A	20040109		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS CASREACT 143:99285

AB Oil-soluble, sulfur scavengers or converts are disclosed where the scavengers include substantially monomeric aldehyde-amine adducts from the reaction of at least one sterically hindered primary or secondary amine and a molar excess of at least one aldehyde or a donor thereof. Preferably the adduct is a compound of formulas CH<sub>2</sub>RNR<sub>1</sub>R<sub>2</sub>, CH<sub>2</sub>RR<sub>4</sub>NR<sub>3</sub>NR<sub>5</sub>CH<sub>2</sub>R or mixts. thereof: where R is a H or a carbon-containing group, R<sub>1</sub> and R<sub>2</sub> are the same or different, at least one being a sterically hindered carbon-containing group having between about 3 and about 24 carbon atoms or R<sub>1</sub> and R<sub>2</sub> can form a ring system, R<sub>3</sub> is a divalent sterically hindered carbon-containing group, R<sub>4</sub> and R<sub>5</sub> are the same or different and are H or a CH<sub>2</sub>R group and where one or more of the carbon atoms of R, R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, R<sub>4</sub>, R<sub>5</sub> or mixts. thereof can be replaced by oxygen atoms in the form of ether moieties, nitrogen groups in the form of tertiary amine or amide moieties or mixts. thereof, and where one or more hydrogen atoms of R, R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, R<sub>4</sub>, R<sub>5</sub> or mixts. thereof can be replaced by fluorine atoms, chlorine atoms or mixts. thereof. Methods are also disclosed for reducing, reducing below a given level or eliminating noxious sulfur species from fluids using these scavengers and for making them. A plot shows pressure and H<sub>2</sub>S concentration verses time for a scavenger embodying this invention compared with comparative triazine scavengers tested at high pressure in a sour gas-drilling mud system pressure and headspace H<sub>2</sub>S composition profiles at about a 10:1 scavenger to H<sub>2</sub>S ratio.

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 10 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2004:355004 CAPLUS  
 DN 140:340168  
 TI Flame retardant compositions containing sterically hindered amine stabilizers  
 IN Kaprinidis, Nikolas; Lelli, Nicola  
 PA Ciba Specialty Chemicals Holding Inc., Switz.  
 SO PCT Int. Appl., 101 pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2004035671	A1	20040429	WO 2003-EP11185	20031009
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
	US 20040097620	A1	20040520	US 2003-675157	20030930
	US 7109260	B2	20060919		
	CA 2501384	A1	20040429	CA 2003-2501384	20031009
	AU 2003282026	A1	20040504	AU 2003-282026	20031009
	EP 1554336	A1	20050720	EP 2003-773639	20031009
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
	CN 1705705	A	20051207	CN 2003-80101433	20031009
	CN 100439434	C	20081203		
	JP 2006503138	T	20060126	JP 2004-544120	20031009
	TW 291478	B	20071221	TW 2003-92128535	20031015
FRAI	US 2002-419260P	P	20021017		
	WO 2003-EP11185	W	20031009		

OS MARPAT 140:340168

AB Organic polymeric substrates, for example polyolefins such as polypropylene, can be made flame retardant by the incorporation of a synergistic mixture of (i) at least one sterically hindered amine stabilizer, (ii) at least one conventional flame retardant selected from the group consisting of the organohalogen, phosphorous containing, isocyanurate and melamine based flame retardants and (iii) at least one acid scavenger. The comps. of the invention combine good flame retardant properties with light stability and good mech. properties. Polyolefin molded articles are stabilized against light, heat and oxygen and made flame retardant with the incorporation of at least one sterically hindered amine and at least one conventional flame retardant, while allowing normally high levels of flame-retardant fillers to be greatly reduced or eliminated.

OSC.G 6 THERE ARE 6 CAPLUS RECORDS THAT CITE THIS RECORD (6 CITINGS)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 11 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2004:108785 CAPLUS  
 DN 141:332890  
 TI Flame retardant and UV stabilized compositions  
 AU Anon.  
 CS Switz.  
 SO IP.com Journal (2003), 3(11), 5 (No. IPCOM000019831D), 1 Oct 2003  
 CODEN: IJPOBX; ISSN: 1533-0001  
 PB IP.com, Inc.  
 DT Journal; Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	IP 19831D		20031001	IP 2003-19831D	20031001
PRAI	IP 2003-19831D		20031001		
AB	A TPO-TPE-TFV (automotive) skin, which can be extruded, calendered, slush-molded or thermoformed, composed of a polyolefin resin and a sterically hindered amine ether flame retardant exhibits excellent flame retardant properties and UV resistance. Another example for combining excellent properties in respect of flame retardancy and UV stability is an expanded polyolefin such as polyethylene (EPE) or expanded polypropylene (EPP) or expanded polyolefin foam, which can be extruded, comprising a sterically hindered amine ether.				



L3 ANSWER 12 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2003:631108 CAPLUS  
 TI Selective oxidation of 2,6-dimethylphenol to polyphenylene ether using  
 copper coordination compounds  
 AU Reedijk, Jan; Gamez, Patrick  
 CS Leiden Institute of Chemistry, Leiden University, Leiden, 2300 RA, Neth.  
 SO Abstracts of Papers, 226th ACS National Meeting, New York, NY, United  
 States, September 7-11, 2003 (2003), COLL-305 Publisher: American Chemical  
 Society, Washington, D. C.  
 CODEN: 69EKY9  
 DT Conference; Meeting Abstract  
 LA English  
 AB The engineering plastic polyphenylene ether (PPE) is commonly  
 produced from 2,6-dimethylphenol and dioxygen, under alkaline conditions with  
 Cu amine compds. as catalysts (see Figure); also many other  
 oxidns. catalyzed by copper coordination compds. and in biol. Cu systems  
 are known. The major technol. problems in the PPE process are the highly  
 colored side product diphenoquinone, water poisoning and the control of  
 the mol. weight Using sterically hindered amine  
 ligands, proper solvent choice and bilayered solvents, these problems have  
 been tackled and reduced. A short overview of the most recent findings  
 and the mechanistic implications will be discussed.

L3 ANSWER 13 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2003:435329 CAPLUS  
 DN 139:7697  
 TI Hindered amine ether flame retardant polyolefin resin pre-expanded  
 particles and in-mold foamed articles prepared therefrom  
 IN Stuart, John Bready; Skarke, Steven Charles; Ogita, Tetsuya; Yamaguchi,  
 Takema; Iwamoto, Tomonori  
 PA Kaneka Corporation, Japan; Kaneka Texas Corporation  
 SO U.S. Pat. Appl. Publ., 12 pp.  
 CODEN: USXXCO  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 20030105194	A1	20030605	US 2001-998170	20011203
	US 6822023	B2	20041123		
	WO 2003048239	A1	20030612	WO 2002-JP12565	20021129
	W: CN, JP, SG				
	RW: BE, DE, FR, GB				
	EP 1454947	A1	20040908	EP 2002-783720	20021129
	EP 1454947	B1	20100217		
	R: BE, DE, FR, GB				
	CN 1599771	A	20050323	CN 2002-824180	20021129
	CN 1309768	C	20070411		
PRAI	US 2001-998170	A	20011203		
	WO 2002-JP12565	W	20021129		

# ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB Flame retardant polyolefin pre-expanded particles made of a resin composition  
 comprising a polyolefin resin and a sterically hindered  
 amine ether flame retardant, which can be molded with  
 good moldability to give in-mold foamed articles which have an excellent  
 flame resistance and do not generate harmful gas at the time of burning.

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 14 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2002:300134 CAPLUS

DN 137:170290

TI A molecular study towards the interaction of phenolic anti-oxidants, aromatic amines and HALS stabilizers in a thermo-oxidative ageing process

AU Barret, Julien; Gijsman, Pieter; Swagten, Josefien; Lange, Ronald F. M.

CS DSM Research, Geleen, 6160 MD, Neth.

SO Polymer Degradation and Stability (2002), 76(3), 441-448

CODEN: PDSTDW; ISSN: 0141-3910

PB Elsevier Science Ltd.

DT Journal

LA English

AB The mol. interactions of phenolic antioxidants, aromatic amines, and HALS stabilizers are studied by heating the stabilizers under oxidative conditions in polar and non-polar solvents. The polar solvent bis(2-methoxyethyl) ether is used to mimic polar engineering plastics like e.g. TPE-U's, whereas the non-polar solvent squalane or 2,6,10,15,19,23-hexamethyltetracosane is used to mimic polypropylene. The oxidation rate is followed by the anal. of samples taken in time using various anal. techniques as e.g. IR, HPLC-PDA, GC-FID, GC-MS, and LC-MS. A general occurring interaction between sterically hindered phenols and aromatic amines, i.e. regeneration of the aromatic amine by the sterically hindered phenol, is demonstrated by varying the mol. structure of the phenol as well as the polarity of the system. Studies using mixts. of a HALS with structurally different sterically hindered phenols visualized a general antagonistic effect between the phenols and the HALS, in which the HALS consumes the phenol. In all stabilizer combinations using the sterically hindered phenols studied here, a stabilization of the resulting quinone form is observed which can, dependent on the mol. structure of this quinone, lead to solubility issues in polymers.

OSC.G 11 THERE ARE 11 CAPLUS RECORDS THAT CITE THIS RECORD (11 CITINGS)

RE.CNT 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 15 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2002:1646 CAPLUS

DN 136:310551

TI The interaction of a phenolic anti-oxidant and an aromatic amine in a thermo-oxidative ageing process

AU Barret, Julien; Gijssman, Pieter; Swagten, Josefien; Lange, Ronald F. M. DSM Research, Geleen, 6160 MD, Neth.

SO Polymer Degradation and Stability (2002), 75(2), 367-374

CODEN: PDSTDW; ISSN: 0141-3910

PB Elsevier Science Ltd.

DT Journal

LA English

AB An interaction of the sterically hindered phenolic antioxidant 1,3,5-trimethyl-2,4,6-Tris(3,5-di-t-butyl-4-hydroxybenzyl)-benzene, 1a, with the aromatic amine 4,4'-bis( $\alpha$ , $\alpha$ -dimethyl-benzyl)diphenylamine, 2a, in co-poly(ether esters) has been observed. This interaction is successfully analyzed by heating 1a and/or 2a in the polyether-mimicking solvent bis(2-methoxyethyl)ether using various anal. techniques. As well as a detailed insight of the action of the individual antioxidants, it has been shown that the quinone structure of the oxidized sterically hindered phenolic antioxidants are only stable in the absence of radical species, which results for the 1a-2a system in the stabilization of the oxidized 1a.

OSC.G 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 16 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2001:886344 CAPLUS

DN 136:38282

TI Aging-resistant epoxy-resin casting compositions, molded articles and their use

IN Bogner, Georg; Brunner, Herbert; Hoehn, Klaus

PA Osram Opto Semiconductors GmbH &amp; Co. Ohg, Germany

SO PCT Int. Appl., 26 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001092396	A1	20011206	WO 2001-DE1848	20010516
	W: CA, CN, JP, KR, US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	DE 10027206	A1	20011213	DE 2000-10027206	20000531
	EP 1287063	A1	20030305	EP 2001-944932	20010516
	EP 1287063	B1	20050316		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	CN 1432040	A	20030723	CN 2001-810252	20010516
	CN 1216933	C	20050831		
	JP 2003535170	T	20031125	JP 2002-500599	20010516
	CN 1706886	A	20051214	CN 2005-10085912	20010516
	TW 260331	B	20060821	TW 2001-90112884	20010529
	US 20030144414	A1	20030731	US 2002-296848	20021127
	US 7183661	B2	20070227		
PRAI	DE 2000-10027206	A	20000531		
	CN 2001-810252	A3	20010516		
	WO 2001-DE1848	W	20010516		

## ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS MARPAT 136:38282

AB A 2-component casting and potting resin composition for assembling and encapsulating automotive, electronic and optoelectronic parts, modules and components for outdoor use, especially LEDs, and suitable for surface mount technol. (SMT), comprises resin component A containing sterically hindered amine as photochem. aging stabilizer, and curing component B which is an acid anhydride hardener. For example, a title composition cured with partially esterified methylhexahydrophthalic anhydride comprised Rutapox VE 3748 (a bisphenol A diglycidyl ether), Byk A-506, Silane A-187 (adhesion enhancer), optical brightener and 0.2% Tinuvin 123 (photochem. stabilizer).

OSC.G 3 THERE ARE 3 CAPLUS RECORDS THAT CITE THIS RECORD (3 CITINGS)

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 17 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 2000:470611 CAPLUS  
 DN 133:135742  
 TI Process for manufacture of stable polyolefins  
 IN Zhang, Baoqing; Jing, Zhenhua; Hong, Xiaoyu  
 PA China Petrochemical Corp., Peop. Rep. China  
 SO Faming Zhuanli Shenqing Gongkai Shuomingshu, 9 pp.  
 CODEN: CNXXEV  
 DT Patent  
 LA Chinese  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CN 1231292	A	19991013	CN 1998-101246	19980403
	CN 1115349	C	20030723		
PRAI	CN 1998-101246		19980403		

OS MARPAT 133:135742

AB The stable polyolefin is produced by catalytic polymerization of C5-12  $\alpha$ -olefin in the presence of solid catalyst, alkylaluminum co-catalyst, phenol-type antioxidant, and/or sterically hindered amine. The solid catalyst is composed of  $\text{TiCl}_4$ , 8.0-15.0% ester and ether internal electron donor compound, and  $\text{MgCl}_2$  carrier; the contents of Mg and Ti in the solid catalyst are 15-25% and 1.0- 5.0%, resp., and the ratio of ether to ester is 0.6-2.8. The ester is  $\text{R}_3\text{OOC-C(R}_1\text{)=C(R}_2\text{)-COOR}_4$  ( $\text{R}_1\text{-R}_4$  = C1-10 alkyl, aryl, or arylalkyl), preferably diisobutyl phthalate, and the ether is  $\text{R}_3\text{O-CH}_2\text{-C(R}_1\text{)(R}_2\text{)-CH}_2\text{-OR}_4$ , preferably 2,2-di-tert-butyl-1,3-dimethoxypropane.  $\text{R}_1\text{R}_2\text{Si(OR)}_2$  external electron donor compound may be added in the polymerization process, and the mole ratio of Al to Si is 30-40. The phenol-type antioxidant is 2,3,5,6-tetra(R)-4-R'-phenol ( $\text{R}$  = H, C1-30 alkyl, C3-30 cycloalkyl, C6-30 aryl, C7-30 arylalkyl, or C1-30 alkoxy; and  $\text{R}'$  =  $\text{CHR}_2$ , or OR), preferably pentaerythritol tetrakis[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propanoate], and the ratio of antioxidant to the polymer is 0.01-0.9%. The ratio of photostabilizer to the polymer is 0.01-0.9%.

L3 ANSWER 18 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2000:387633 CAPLUS

DN 133:164354

TI Synthesis of Novel Hindered Amine Light Stabilizers (HALS) and Their Copolymerization with Ethylene or Propylene over Both Soluble and Supported Metallocene Catalyst Systems

AU Wilen, Carl-Eric; Auer, Markku; Stranden, Juha; Naesman, Jan H.; Rotzinger, Bruno; Steinmann, Alfred; King, Roswell E., III; Zweifel, Hans; Drewes, Rolf

CS Laboratory of Polymer Technology, Abo Akademi University, Turku, FIN-20500, Finland

SO Macromolecules (2000), 33(14), 5011-5026

CODEN: MAMOBX; ISSN: 0024-9297

PB American Chemical Society

DT Journal

LA English

AB Novel polymerizable hindered amine light stabilizers

(HALS) such 1-(but-3-enyl)-2,2,6,6-tetramethylpiperidine (1), 1-(undec-10-enyl)-2,2,6,6-tetramethylpiperidine (2), 4-(but-3-enyl)-1,2,2,6,6-pentamethyl-3,4-dehydropiperidine (3), 2-(but-3-enyl)-2,2,6,6-trimethylpiperidine (4), 4-(but-3-enyl)-1,2,2,6,6-pentamethyl-4-piperidyl ether (5), 4-(undec-10-enylamide)-1,2,2,6,6-pentamethylpiperidine (6), 4-((N-n-butyl)-undec-10-enylamide)-1,2,2,6,6-pentamethylpiperidine (7), and bis((N-n-butyl)-N-2,2,6,6-tetramethylpiperidine)-N-n-butyl-N-allyltriazine (8) were synthesized. All the aforementioned HALS monomers except for 5 and 8 were successfully copolymerized in fair to high yields with ethylene or propylene over 8 different group 4 metallocene catalysts using methylalumoxane (MAO) as cocatalyst. Copolymers were also performed over a supported metallocene/SiO<sub>2</sub>/MAO/triisobutylaluminum(TIBA) catalyst system. The silica-supported metallocene catalyst system readily promoted copolymerization of the sterically hindered monomer 2 with ethylene, although copolymers using either 6 or 7 as comonomer failed. Moreover, a catalyst derived from the reaction of rac-[dimethylsilylenebis(1-indenyl)]zirconium dichloride (CA1) with Et<sub>3</sub>Al and trityl tetra(perfluorophenyl)borate (TRI-FABA) afforded HALS copolymers in high yields. Surprisingly, TRI-FABA, a strong Lewis acid, could impede the Lewis base activity of HALS monomers such as 2, 6 and 7 provided a sufficient relative amount of TRI-FABA was employed. Thus, once an equilibrium concentration between TRI-FABA and HALS monomer was established, the

presence of HALS monomer no longer affected the rate of polymerization

Normally,

metallocene catalysts are severely poisoned when traces of polar monomers (Lewis bases) are present, due to the Lewis acidic nature of the catalyst. Furthermore, a series of standard ethylene homopolymers over rac-[dimethylsilylenebis(4,5,6,7-tetrahydro-1-indenyl)]zirconium dichloride (CA2)/MAO catalyst system was performed in the presence of different sterically hindered amine model compounds such as 1-(1-methylene-2,6-di-tert-butylphenol)-2,2,6,6-tetramethylpiperidine (A), N,N-diisopropylaniline (B), 1-octyl-2,2,6,6-tetramethylpiperidine (C), 1-benzyl-2,2,6,6-tetramethylpiperidine (D), 2,2,6,6-tetramethylpiperidine (E), 1,2,2,6,6-pentamethylpiperidine (F), diisopropylethylamine (G), 1,2,2,6,6-pentamethyl-4-oxopiperidine (H), 1,2,2,6,6-tetramethylpiperidine-1-oxyl (I), 1-propargyl-2,2,6,6-tetramethylpiperidine (J), 4-N,N-bis(n-butylamino)-2,2,6,6-tetramethylpiperidine (K), tris(N-butyl-N-2,2,6,6-tetramethylpiperidineamino)triazine (L), and tris(dibutylamino)triazine (M). Some of the amine model compounds.

are highly reactive and deactivating whereas others are less so. Much preferred are those HALS structures which have a sterically demanding substituent attached on N and no addnl. heteroatoms in 4-position of the piperidine ring in terms of metallocene/MAO catalyst activity. <sup>13</sup>C NMR analyses revealed that the produced materials are random copolymers containing isolated HALS branches and that the propylene copolymers have highly stereoregular microstructures. According to size exclusion chromatog., the copolymers have mol. weight distributions close to 2, which are characteristic for polymers produced over single-site catalysts. The copolymers contained 0.2-14.1 weight-% of HALS units and exhibited high UV and thermooxidative stabilities even after exhaustive extraction with a mixture of refluxing (50:50) cyclohexane/2-propanol. For example, the poly(ethylene-co-4) copolymer with a HALS content of 0.2 weight-% exhibited considerable improved thermooxidative stability in comparison to unstabilized polyethylene, i.e., for the copolymer the carbonyl peak had not appeared after one year of oven aging at 115°, whereas unstabilized polyethylene shows a strong increase in the carbonyl index within 2 days.

OSC.G 14 THERE ARE 14 CAPLUS RECORDS THAT CITE THIS RECORD (14 CITINGS)  
RE.CNT 63 THERE ARE 63 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT



L3 ANSWER 19 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 2000:371854 CAPLUS

DN 132:335883

TI Compositions containing acrylic and indene resins for coatings for wood, masonry, and metals

IN Jurcau, Dorin; Gherdan, Mircea; Tamas, Ladislau; Drutau, Mihaela

PA S.C. "Azur" S.A., Timisoara, Rom.

SO Rom., 4 pp.

CODEN: RUXXA3

DT Patent

LA Romanian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	RO 110523	B1	19960130	RO 1991-146941	19910218
PRAI	RO 1991-146941		19910218		

AB Glossy coatings with good resistance to water and salt solns. for the title substrates are manufactured from compns. containing 50% acrylic resin-PhMe

solution 15-35, pigment and fillers 15-32, pigment dispersants 0.2-2, 50% indene resin-PhMe solution 30-60, BuOAc 1-4, ethylene glycol monoethyl ether acetate 0.3-3, solvent naphtha 1-50, and sterically hindered amine and oxalamide UV absorber 0.1-1 parts.

The acrylic resin is manufactured by polymerization of Me methacrylate 3-25, styrene

20-60, Bu acrylate 10-50, Bu methacrylate 3-15, 2-ethylhexyl acrylate 10-40,  $\alpha$ -methylstyrene 3-20, and acrylic acid 1-10 parts.

L3 ANSWER 20 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1999:205371 CAPLUS  
 DN 130:237281  
 TI Preparation of fluoromethyl ethers by the fluorination of chloromethyl  
 ethers with sterically hindered tertiary amine hydrofluoride salts  
 IN Kudzma, Linas V.; Lessor, Ralph A.; Rozov, Leonid A.; Ramig, Keith  
 PA Baxter International Inc., USA  
 SO U.S., 4 pp.  
 CODEN: USXXAM  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5886239	A	19990323	US 1997-975689	19971121
	TW 472035	B	20020111	TW 1998-87118992	19981117
	CA 2277928	A1	19990603	CA 1998-2277928	19981118
	CA 2277928	C	20080610		
	WO 9926910	A1	19990603	WO 1998-US24746	19981118
	W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW				
	RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	AU 9914205	A	19990615	AU 1999-14205	19981118
	AU 752881	B2	20021003		
	EP 958269	A1	19991124	EP 1998-958096	19981118
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE, FI				
	BR 9806786	A	20000509	BR 1998-6786	19981118
	HU 2000003190	A2	20010528	HU 2000-3190	19981118
	HU 2000003190	A3	20011128		
	JP 2001508468	T	20010626	JP 1999-529114	19981118
	JP 4355369	B2	20091028		
	RU 2169724	C2	20010627	RU 1999-118578	19981118
	EP 1277724	A1	20030122	EP 2002-79024	19981118
	EP 1277724	B1	20050629		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE, FI				
	CN 1140488	C	20040303	CN 1998-801928	19981118
	AT 298738	T	20050715	AT 2002-79024	19981118
	ES 2241953	T3	20051101	ES 2002-79024	19981118
	PL 191789	B1	20060731	PL 1998-334702	19981118
	ZA 9810627	A	19990525	ZA 1998-10627	19981120
	MX 9906614	A	20000228	MX 1999-6614	19990715
PRAI	US 1997-975689	A	19971121		
	EP 1998-958096	A3	19981118		
	WO 1998-US24746	W	19981118		

## ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS CASREACT 130:237281; MARPAT 130:237281

AB Fluoromethyl ethers R1(R2)C(A)OCH2F [A = lower fluoroalkyl, fluorine, chlorine; R1, R2 = hydrogen, (un)branched lower alkyl, lower fluoroalkyl, fluoro, chloro; such that  $\geq 1$  of A, R1, R2 = lower fluoroalkyl, (un)branched lower alkyl] [e.g., 2,2,2-trifluoro-1-(trifluoromethyl)ethyl ether], useful as inhalation anesthetics, are prepared by the fluorination of the corresponding chloromethyl ether [e.g., 2-(chloromethoxy)-1,1,1,3,3,3-hexafluoropropane] with a sterically hindered tertiary amine hydrofluoride salt (e.g., diisopropylethylamine hydrofluoride).

10/591,778

OSC.G	8	THERE ARE 8 CAPLUS RECORDS THAT CITE THIS RECORD (8 CITINGS)
RE.CNT	17	THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD
		ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 21 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1998:701048 CAPLUS

DN 129:317691

OREF 129:64797a

TI Polyurethane coating material, method for its preparation and application as coating varnish or clear varnish, especially for coating of synthetic materials

IN Hintze-Bruening, Horst

PA Basf Coatings A.-G., Germany

SO Ger., 16 pp.

CODEN: GWXXAW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19723504	C1	19981015	DE 1997-19723504	19970605
	CA 2289510	A1	19981210	CA 1998-2289510	19980602
	WO 9855526	A1	19981210	WO 1998-EP3285	19980602
	W: BR, CA, JP, KR, US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	EP 986595	A1	20000322	EP 1998-929411	19980602
	EP 986595	B1	20020417		
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE, PT				
	JP 2002502458	T	20020122	JP 1999-501485	19980602
	AT 216407	T	20020515	AT 1998-929411	19980602
	ES 2175725	T3	20021116	ES 1998-929411	19980602
	BR 9810085	A	20000808	BR 1998-10085	19981210
	US 6297314	B1	20011002	US 2000-424092	20000112
PRAI	DE 1997-19723504	A	19970605		
	WO 1998-EP3285	W	19980602		

## ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB Polyurethane coatings with good resistance to weather, chems., moisture, and impact for plastics are manufactured from compns. containing  $\geq 1$  polyester with OH number 80-200 mg KOH/g and acid number  $< 10$  mg KOH/g,  $\geq 1$  polyacrylate with OH number 80-200 mg KOH/g and acid number  $< 20$  mg KOH/g,  $\geq 1$  di- and(or) polyisocyanate having (un)blocked NCO groups,  $\geq 1$  UV absorber,  $\geq 1$  sterically hindered amine having amino ether groups as light stabilizer, and  $\geq 1$  solvent.

OSC.G 3 THERE ARE 3 CAPLUS RECORDS THAT CITE THIS RECORD (3 CITINGS)

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 22 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1998:639448 CAPLUS

DN 129:343551

OREF 129:69977a,69980a

TI Zirconium-catalyzed and zirconium-promoted cyclization reactions of non-conjugated dienes with alkylmagnesium halides to give cycloalkylmethylmagnesium derivatives

AU Negishi, E.; Rousset, Christophe J.; Choueiry, Daniele; Maye, John P.; Suzuki, Noriyuki; Takahashi, Tamotsu

CS Dep. Chem., Purdue Univ., West Lafayette, IN, 47907, USA

SO Inorganica Chimica Acta (1998), 280(1-2), 8-20

CODEN: ICHAA3; ISSN: 0020-1693

PB Elsevier Science S.A.

DT Journal

LA English

OS CASREACT 129:343551

AB The stoichiometric reaction of certain non-conjugated dienes with n-BuZrCp<sub>2</sub> provides the corresponding zirconabicycles, such as trans-3-bis(cyclopentadienyl)zirconabicyclo[3.3.0]octane, that can be fully characterized by spectroscopic means. Their treatment with EtMgBr or n-BuMgCl in THF gives the corresponding monocyclic monomagnesium derivs. along with the corresponding alkene-ZrCp<sub>2</sub> derivs. in high yields. In cases where the Grignard reagent is either sterically hindered or lacking  $\beta$ -H, little or no reaction may occur, although some, e.g., s-BuMgCl, react, albeit slowly, to give the expected products in high yields. In cases where either a Grignard reagent in di-Et ether or a dialkylmagnesium (irresp. of solvent) is used, the major product is the corresponding dimagnesium derivative. A couple of intramol. transmetalation paths are proposed for these cases. The Cp<sub>2</sub>ZrCl<sub>2</sub>-catalyzed reaction of 1,6-heptadiene with EtMgBr fails to induce the desired bicyclization-ring opening sequence to give the corresponding monocyclic monomagnesium derivs. On the other hand, the corresponding reaction with n-BuMgBr does proceed as desired to give the monocyclic monomagnesium product which contains a minor amount of the corresponding exo-methylene derivative. Only traces, if any, of the corresponding dimagnesium derivs. reported to be the major products in Et<sub>2</sub>O are formed. This procedure has been applied to catalytically convert several other dienes, i.e., (E)-1-phenyl-1,6-heptadiene, 2,4,4-trimethyl-1,6-heptadiene, diallyl(benzyl)amine, 1,7-octadiene and 1,2-diallylbenzene, into the corresponding monocyclized compds. in moderate to excellent combined yields.

OSC.G 22 THERE ARE 22 CAPLUS RECORDS THAT CITE THIS RECORD (22 CITINGS)

RE.CNT 63 THERE ARE 63 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 23 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1998:151193 CAPLUS

DN 128:168827

OREF 128:33257a,33260a

TI Stable polyorganosiloxane-based compositions with crosslinkable functional groups and their use for producing antiadhesive coating

IN Priou, Christian; Soldat, Andre; Kerr, Stuart R., III; Beaty, Reeshemah

PA Rhone-Poulenc Chimie SA, Fr.; Priou, Christian; Soldat, Andre; Kerr,

Stuart R., III; Beaty, Reeshemah

SO PCT Int. Appl., 30 pp.

CODEN: PIXXD2

DT Patent

LA French

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9807798	A1	19980226	WO 1997-FR1492	19970814
	W: AL, AU, BA, BB, BG, BR, CA, CN, CU, CZ, EE, GE, HU, IL, IS, JP, KP, KR, LC, LK, LR, LT, LV, MG, MK, MN, MX, NO, NZ, PL, RO, SG, SI, SK, TR, TT, UA, US, UZ, VN, YU, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
	FR 2752582	A1	19980227	FR 1996-10330	19960821
	FR 2752582	B1	20030613		
	CA 2264013	C	19980226	CA 1997-2264013	19970814
	CA 2264013	A1	19980226		
	AU 9740189	A	19980306	AU 1997-40189	19970814
	AU 732617	B2	20010426		
	EP 920483	A1	19990609	EP 1997-937629	19970814
	EP 920483	B1	20020213		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO				
	JP 2000516660	T	20001212	JP 1998-510462	19970814
	JP 4190585	B2	20081203		
	AT 213263	T	20020215	AT 1997-937629	19970814
	ES 2168660	T3	20020616	ES 1997-937629	19970814
	PT 920483	E	20020628	PT 1997-937629	19970814
	US 6218445	B1	20010417	US 2000-242713	20000110
PRAI	FR 1996-10330	A	19960821		
	WO 1997-FR1492	W	19970814		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS MARPAT 128:168827

AB The title radiation-crosslinkable, storage-stable composition is based on  $\geq 1$  polyorganosiloxane with crosslinkable epoxy or vinyl ether functional groups, and contains a stabilizing amine agent, and an initiator system for hardening under radiation, in particular UV radiation. The amine is a secondary or tertiary amine, a sterically hindered cyclic amine, or an amine consisting of both types of groups.

A composition contained TMS-terminated (epoxycyclohexyl)ethyl Me, di-Me siloxane, ditolylidinium tetrakis(pentafluorophenyl)borate, and trioctylamine.

OSC.G 8 THERE ARE 8 CAPLUS RECORDS THAT CITE THIS RECORD (9 CITINGS)

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 24 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1997:234099 CAPLUS

DN 126:225981

OREF 126:43703a,43706a

TI Preparation of heat-resistant polyurethane-polyurea foam

IN Dempsey, Michael P.

PA Bayer A.-G., USA

SO Can. Pat. Appl., 29 pp.

CODEN: CPXXEB

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CA 2172678	A1	19961118	CA 1996-2172678	19960326
PRAI	US 1995-442624	A	19950517		

AB Title foam is prepared by reacting, at isocyanate index 100-115, (a) an organic polyisocyanate with (b) 40-65 weight%, based on the total amount of components (b), (c), and (d), of an isocyanate-reactive compound containing  $\geq 2$  isocyanate-reactive groups and having a number average mol. weight of 400-10,000;

(c) 10-35 weight%, based on total of (b), (c), and (d), of an isocyanate-reactive compound containing  $\geq 2$  isocyanate-reactive groups other than amino groups and having a mol. weight of 32-399; (d) 5-35 weight%, based on total of (b), (c), and (d), of an organic amine containing  $\geq 1$  amino group having a moderated reactivity toward the organic polyisocyanate, as indicated by a gel time  $> \text{apprx.} 5 \text{ s}$  measured from the material initiation time at 25-40°C, and number average mol. weight 86-400, selected from (1) sterically hindered aromatic amines in which  $\geq 1$  aromatic ring substituents are ortho to the amino groups, (2) aromatic amines other than amines (d)(1) in which  $\geq 1$  of the amino groups exhibits reduced reactivity due primarily to electronic effects rather than steric factors, (3) amines having secondary amino groups, (4) nonarom. amines having sterically hindered primary amino groups, and (5) mixts. thereof; and (e) optionally, rigid fibers. The products are useful for automotive applications (no data). Thus, Mondur 489 containing 34.9 parts milled glass fiber/100 parts polyol was reacted with a polyol composition comprising ethylene glycol 18, polyethylene glycol-polypropylene glycol glyceryl ether 55, and polypropylene oxide ether with ethylenediamine 3 parts, containing 20 parts Ethacure 300 to form a plaque having d. 0.5 g/cm<sup>3</sup>, flexural modulus 865 MPa, and parallel heat sag 5.3 mm and perpendicular heat sag 8.5 mm (6-in. overhang, 121°, 1 h), compared with 0.5, 779, 20.5, and 27.5, resp., for a sample without the Ethacure 300.

L3 ANSWER 25 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1995:994682 CAPLUS

DN 124:10548

OREF 124:2191a,2194a

TI Elastic amine-modified epoxy resin composition, especially suitable as one-component adhesive

IN Marten, Manfred; Wehner, Bernhard

PA Hoechst A.-G., Germany

SO Eur. Pat. Appl., 18 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 675185	A2	19951004	EP 1995-104296	19950323
	EP 675185	A3	19960110		
	R: AT, BE, CH, DE, DK, ES, FR, GB, IE, IT, LI, NL, PT, SE				
	DE 4410786	A1	19951005	DE 1994-4410786	19940328
	CA 2145589	A1	19950929	CA 1995-2145589	19950327
	JP 08034833	A	19960206	JP 1995-68175	19950327
PRAI	DE 1994-4410786	A	19940328		

AB The title composition contains a reaction product of a polyepoxide, a polyoxyalkylene amine, and, optionally, a polycarboxylic acid, a reaction product of a polyepoxide and a sterically hindered amine, other epoxides, hardeners, and additives and is especially useful as a 1-component adhesive for bonding steel parts in the manufacture of automobiles. A reaction product of a bisphenol A epoxy resin and Jeffamine M 600, a reaction product of polypropylene glycol diglycidyl ether (Beckopox EP 075) and 2-aminobutane, dicyandiamide, and silica were used in an adhesive for bonding steel plates.



L3 ANSWER 26 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1994:272068 CAPLUS

DN 120:272068

OREF 120:48195a,48198a

TI Chemical strategies for compatibilization of immiscible polymer blends: polyphenylene ether-nylon copolymer formation through nucleophilic displacement on aryloxytriazine-capped polyphenylene ethers

AU Brown, S. Bruce

CS Polym. Chem. Lab., Gen. Electr. Res Dev. Cent., Schenectady, NY, 12301, USA

SO Polymer Preprints (American Chemical Society, Division of Polymer

Chemistry) (1992), 33(2), 598-9

CODEN: ACPPAY; ISSN: 0032-3934

DT Journal

LA English

AB The OH end-groups of poly(phenylene ether) (PPE) were functionalized by 3 different methods, i.e., chloride displacement from a chlorodiarlyoxytriazine, transesterification with a tris(aryloxytriazine) and branching and/or chain-extension with a dichlorotriazine or with cyanuric chloride. Coextrusion of the aryloxytriazine-functionalized PPE with amine-terminated nylons resulted in PPE-nylon copolymer formation through nucleophilic displacement of an aryloxy group from the triazine-functionalized PPE by the amine end-groups of the nylon. The resulting compatibilized blends had excellent mech. properties. The displacement to form PPE-nylon copolymer competed with the displacement to form triazine end-capped nylon and no copolymer. The sterically hindered 2,6-dimethylphenoxy group of PPE was probably more difficult to displace during melt extrusion than an unhindered phenoxy group on the triazine ring.

OSC.G 7 THERE ARE 7 CAPLUS RECORDS THAT CITE THIS RECORD (7 CITINGS)

L3 ANSWER 27 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1994:135387 CAPLUS

DN 120:135387

OREF 120:23881a,23884a

TI Amine-modified metathesis catalysts for production of polymers and copolymers of cycloalkene of norbornene type

IN Stohandl, Jiri; Vozka, Pavel; Varekova, Irena; Krafat, Miroslav; Ondruj, Jiri; Mejzlik, Jiri; Balcar, Hynek; Stpanek, Kamil; Heller, Gerhard; Lederer, Jaromir

PA Chemopetrol, S.P. Litvinov Vyzkumny Ustav Makromolekularni Chemie, Czech.

SO PCT Int. Appl., 34 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9312158	A1	19930624	WO 1992-CS34	19921216
	W: HU, US				
	RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	CZ 279861	B6	19950712	CZ 1991-3808	19911216
	SK 278739	B6	19980204	SK 1991-3808	19911216
	EP 571579	A1	19931201	EP 1992-924538	19921216
	EP 571579	B1	19980401		
	R: BE, DE, FR, GB, IT, NL				
	US 5455318	A	19951003	US 1993-104168	19931015
PRAI	CS 1991-3808	A	19911216		
	WO 1992-CS34	W	19921216		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

OS MARPAT 120:135387

AB A sterically hindered amine modifies

≥1 W compound containing Cl, Br, and/or I to form catalyst precursor which is used with cocatalyst selected from Al, Sn, Pb, Si, Li, Mg, B, Zn, and/or Ge compds., optionally moderator compds., e.g. ethers, in the manufacture of copolymers of norbornene type by reaction injection molding. WCl<sub>6</sub> was modified with 2,2,6,6-tetramethyl-4-hydroxy-4-aminocarbonylpiperidine at mol ratio 1:1 in PhMe and mixed with dicyclopentadiene (I), then admixed with cocatalyst solution at final mol ratio W component:diethylaluminum chloride:di-Bu ether:I at 1:8:20:2000, and heated at 60° to give polymer.

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 28 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1993:39215 CAPLUS

DN 118:39215

OREF 118:7159a,7162a

TI Natural products. Antitubulin effect of congeners of N-acetylcolchiciny methyl ether: synthesis of optically active 5-acetamidodeaminocolchiciny methyl ether and of demethoxy analogs of deaminocolchiciny methyl ether

AU Boye, O.; Brossi, A.; Yeh, H. J. C.; Hamel, E.; Wegrzynski, B.; Toome, V.

CS Lab. Struct. Biol., Natl. Inst. Health, Bethesda, MD, 20892, USA

SO Canadian Journal of Chemistry (1992), 70(5), 1237-49

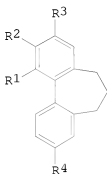
CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

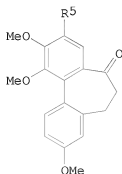
LA English

OS CASREACT 118:39215

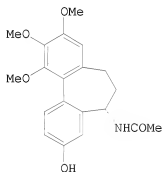
GI



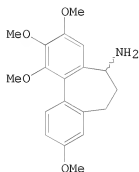
I



II



III



IV

AB Trimethoxy-substituted dihydrodibenzocycloheptenes I (R1 = R2 = R4 = MeO, R3 = H; R1 = R3 = R4 = MeO, R2 = H; R2 = R3 = R4 = MeO, R1 = H; R1 = R2 = R3 = MeO, R4 = H), required for a structure-activity study measuring the inhibition of tubulin polymerization in vitro, were synthesized by four different

routes: (1) Synthesis of I (R1 = R2 = R4 = MeO, R3 = H) was achieved from 2,3-dimethoxybenzaldehyde via 2,3-(MeO)2C6H3C6H3(OMe)CHO-4,2, chain lengthening to propionic acid, acid-catalyzed cyclization toward dihydrodibenzocycloheptene II (R5 = H), and removal of the carbonyl group.

(2) Compound I (R1 = R3 = R4 = MeO, R2 = H) was obtained by eliminating the sterically most hindered methoxy group in the oxime of

II (R5 = MeO) by metal reduction in alc. (3) Compound I (R2 = R3 = R4 = MeO,

R1

= H) was prepared from the corresponding biphenyl aldehyde obtained by Grignard reaction on an oxazoline. (4) Compound I (R1 = R2 = R3 = MeO, R4 = H) was obtained by reductive deoxygenation of N-acetylcolchinol (III) tetrazolyl ether derivative. The key role of the aromatic oxygen atoms in colchicine and allo congeners as points of interaction with the colchicine binding site on tubulin was demonstrated by the lack of inhibitory activity of compds. I. Optically active 5-acetamide isomers of N-acetylcolchinyll Me ether were obtained after chemical resolution of amine IV. The absolute configuration of the optical isomers of IV was determined by 1H NMR and CD measurements. These compds. were found inactive as inhibitors of tubulin polymerization

OSC.G 17 THERE ARE 17 CAPLUS RECORDS THAT CITE THIS RECORD (18 CITINGS)

L3 ANSWER 29 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1992:427623 CAPLUS

DN 117:27623

OREF 117:5014h,5015a

TI The effects of steric hindrance on sub-glass transitions in epoxy polymers

AU Balizer, Edward; Duffy, James V.

CS Nav. Surf. Warf. Cent., Silver Spring, MD, 20903-5000, USA

SO Polymer (1992), 33(10), 2114-22

CODEN: POLMAG; ISSN: 0032-3861

DT Journal

LA English

AB Pairs of sterically hindered and unhindered linear aliphatic and aromatic diamines were prepared and used as curatives for the diglycidyl ether of bisphenol A. The steric hindrance was caused by Me group substitution of a H atom adjacent to the amine . For each pair, the hindered diamine cure had a lower d. and a higher glass transition. Another pair of diamines was prepared for which the Me group was replaced by Et and Bu side chains; for these resins, both the d. and glass transition decreased. Torsional pendulum results showed that the sub-glass transition for the hindered cures shifted to lower temps. and had a greater activation energy. Anal. by the Havriliak-Negami dispersion equation showed that the hindered resins had broader and more sym. relaxations. The background hysteresis loss outside of the relaxation region was analyzed by the Nutting equation and was found to decrease with steric hindrance.

L3 ANSWER 30 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1992:7559 CAPLUS

DN 116:7559

OREF 116:1467a,1470a

TI Manufacture of polyurethane-polyurea and/or polyurea reaction-injection moldings with improved coatability

IN Ueda, Hiroshi; Uchida, Yuji; Yoshida, Yoshio; Inoue, Hiroshi; Kaneda, Toshikazu; Moriya, Toshiaki; Kumazawa, Tsutomu

PA Mitsui Toatsu Chemicals, Inc., Japan

SO Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 03195770	A	19910827	JP 1989-332643	19891225
PRAI	JP 1989-332643		19891225		
OS	MARPAT 116:7559				

AB Title moldings are prepared from polyisocyanate, polyisocyanate-reactive polymer (A) having mol. weight 800-12,000, 5-50% (based on A) sterically hindered aromatic diamine as chain extender, and 0.01-2.0% (based on A) sterically hindered amine-type light stabilizer and possibly other conventional stabilizers. Thus, reaction injection molding a formulation of ethylene oxide-propylene oxide copolymer glycerin ether (OH number 34, containing 85% propylene oxide, having 15% ethylene oxide at ends) 100, an 80:20 mixture of 1-methyl-3,5-diethyl-2,4-diaminobenzene and 1-methyl-3,5-diethyl-2,6-diaminobenzene as chain extenders 25, Dabco 33LV 0.15, DBTDL 0.15, Sanole LS 770 as light stabilizer 0.5, and tripropylene glycol-MDI prepolymer (NCO index 100) 64 g and postcuring 30 min at 120° gave moldings with excellent resistance to discoloration and yellowing.

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

L3 ANSWER 31 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1991:453266 CAPLUS  
 DN 115:53266  
 OREF 115:9201a,9204a  
 TI Antioxidant for lubricants and lubricant compositions  
 IN Evans, Samuel  
 PA Ciba-Geigy A.-G., Switz.  
 SO Eur. Pat. Appl., 38 pp.  
 CODEN: EPXXDW  
 DT Patent  
 LA German  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 406826	A1	19910109	EP 1990-112762	19900704
	EP 406826	B1	19930811		
	R: BE, DE, DK, ES, FR, GB, IT, NL				
	CA 2020558	A1	19910108	CA 1990-2020558	19900705
	CA 2020558	C	20011106		
	BR 9003187	A	19910827	BR 1990-3187	19900705
	DD 297443	A5	19920109	DD 1990-342534	19900705
	JP 03045696	A	19910227	JP 1990-179385	19900706
	JP 2943004	B2	19990830		
	ZA 9005305	A	19910529	ZA 1990-5305	19900706
	CN 1048560	A	19910116	CN 1990-103183	19900707
	CN 1028243	C	19950419		
	KR 151400	B1	19981001	KR 1990-10358	19900707
	US 5268113	A	19931207	US 1992-933599	19920820
PRAI	CH 1989-2528	A	19890707		
	US 1990-546277	B1	19900628		

# ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB The antioxidant contains a sterically hindered amine and a phenol substituted in one or both ortho positions by C1-24 alkyl, C5-12 cycloalkyl, C7-9 phenylalkyl, or a CH2SR1 group, where R1 is specific alkyl, ether chain or Ph, and substituted in the para position by H, C1-18 alkyl, or specific S-, O-, or N-containing chains. The additive is especially suitable for motor oils; it reduces sludge formation significantly.

L3 ANSWER 32 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1990:159257 CAPLUS  
 DN 112:159257  
 OREF 112:26935a,26938a  
 TI Manufacture of polyoxyalkylene alkylamine derivatives  
 IN Gerkin, Richard Michael; Kirchner, David Lee  
 PA Union Carbide Corp., USA  
 SO Eur. Pat. Appl., 7 pp.  
 CODEN: EPXXDW  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 343486	A2	19891129	EP 1989-108795	19890516
	EP 343486	A3	19901114		
	EP 343486	B1	19940629		
	R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, LU, NL, SE				
	US 4960942	A	19901002	US 1988-195010	19880517
	JP 02064126	A	19900305	JP 1989-120623	19890516
	JP 06049754	B	19940629		
	CA 1339907	C	19980609	CA 1989-599756	19890516
PRAI	US 1988-195010	A	19880517		

# ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB The title compds. are prepared from polyethers bearing NH<sub>2</sub> groups by catalytic alkylation with sterically hindered alcs. in the presence of H at 175-250°/250-2000 psi. Heating Jeffamine T-5000 [trimethylolpropane polypropylene glycol ether (1:3) tris(aminopropyl) ether] 1204.1, iso-ProH 424.7, and Ni catalyst 42.1 g with H at 190°/200 psi for 20 h gave a product containing 86% secondary amine; vs. 4% with MeOH instead of iso-ProH.

OSC.G 3 THERE ARE 3 CAPLUS RECORDS THAT CITE THIS RECORD (3 CITINGS)



L3 ANSWER 33 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1988:151730 CAPLUS

DN 108:151730

OREF 108:24921a,24924a

TI Recycling of rigid polyurethane wastes

IN Ionescu, Mihail; Dumitriu, Viorica Tatiana; Mihalache, Ioana; Stoenescu, Felicia; Mihai, Stanca

PA Combinatul Petrochimic Midia, Navodari, Rom.

SO Rom., 4 pp.

CODEN: RUXXA3

DT Patent

LA Romanian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	RO 89944	B1	19860830	RO 1984-114849	19840613
PRAI	RO 1984-114849		19840613		

AB Rigid polyurethane foam wastes are simultaneously aminolyzed by NH<sub>3</sub>, ethylene diamine (I), diethylene triamine, hexamethylenediamine, or ethanolamine and alkoxyated by ethylene oxide, propylene oxide (II), butylene oxide, Ph glycidyl ether, or styrene oxide optionally in the presence of a HO group-containing, sterically hindered tert-amine at aminolyzing amine-waste ratio 1:(1-2) and 160-200° to rapidly give polyols, useful in the manufacture of polyurethane foams. Thus, adding 270 mL II in 1.5 h to a mixture containing 100 g I, 100 g crumbled rigid polyurethane foam waste, and 2 mL dimethylethanolamine catalyst at 160-190° and 4 kg/cm<sup>2</sup> N pressure, and reaction mixture was stirred an addnl. 1 h at 110-115°/1.2 kg/cm<sup>2</sup> to give a brown liquid polyol with OH index 522 mg KOH/g, acid index 1.8 mg KOH/g, and viscosity 5220 cP, from which a polyurethane foam with satisfactory properties could be prepared

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

L3 ANSWER 34 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1987:125716 CAPLUS

DN 106:125716

OREF 106:20437a,20440a

TI Cosmetics containing amide-amine condensates for protecting hair and skin against light

IN Mahieu, Claude; Papantoniou, Christos

PA Oreal S. A. , Fr.

SO Ger. Offen., 13 pp.

CODEN: GWXXBX

DT Patent

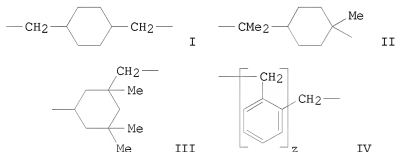
LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3622066	A1	19870108	DE 1986-3622066	19860701
	FR 2584292	A1	19870109	FR 1985-10158	19850703
	FR 2584292	B1	19871016		
	FR 2600249	A2	19871224	FR 1986-8858	19860619
	FR 2600249	B2	19880916		
	BE 905033	A1	19870102	BE 1986-216861	19860702
	NL 8601723	A	19870202	NL 1986-1723	19860702
	GB 2177916	A	19870204	GB 1986-16124	19860702
	GB 2177916	B	19890726		
	US 4844889	A	19890704	US 1986-881279	19860702
	CA 1279262	C	19910122	CA 1986-512929	19860702
	JP 62026211	A	19870204	JP 1986-155223	19860703
PRAI	FR 1985-10158	A	19850703		
	FR 1986-8858	A	19860619		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

GI



AB The title cosmetics contain a saturated condensate obtained by the addition of

a

bisacrylamide  $H_2C:CHCONHCHR_1NHCOCH:CH_2$  ( $R_1 = H$ , C1-4 alkyl) to  $\geq 1$  sterically-hindered diamine  $H_2NRRNH_2$  [ $R = (CH_2)_nCR_2Me(CH_2)_m$ , I, II, III, IV, etc.;  $R_2 = H$ , Me;  $n, m = 0, 1$ ;  $z = 1, 2$ ] followed by saturation (hydrogenation or thiol or amine addition). A saturated polycondensate was prepared by the polyaddn. of (3-aminomethyl-3,5,5-trimethyl)cyclohexylamine with methylenebisacrylamide, followed by saturation with cysteine. A shampoo contained the condensate 1 g, Na ethoxylated C12-14 alkyl ether sulfate 5g, 30% cocoamido Pr betaine 3 g, HCl to pH 7, and water to 100 g.

10/591,778

The condensate increased the mech. strength of bleached human hair.

OSC.G 1        THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (2 CITINGS)

L3 ANSWER 35 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
AN 1987:49589 CAPLUS  
DN 106:49589  
OREF 106:8203a,8206a  
TI Preparation of hindered lithium amide bases and rates of their reaction  
with ether solvents  
AU Kopka, Ihor E.; Fataftah, Zacharia A.; Rathke, Michael W.  
CS Dep. Chem., Michigan State Univ., East Lansing, MI, 48824, USA  
SO Journal of Organic Chemistry (1987), 52(3), 448-50  
CODEN: JOCEAH; ISSN: 0022-3263  
DT Journal  
LA English  
OS CASREACT 106:49589  
AB The conversion of hindered secondary amines EtCR1R2NHCR3R4Et [R1, R2 = Me,  
Et; R1R2 = R3R4 = (CH2)5] to the corresponding lithium amides was examined  
under a variety of conditions. The more hindered secondary amines were  
inert to MeLi, sec-butyllithium, and BuLi in the absence of solvent  
additives. In the presence of Me2NCH2CH2NMe2, all of the amines reacted  
with BuLi at reasonable rates (<24 h, 25°). The lithium amides  
reacted fairly rapidly with THF solvent (T1/2 < 10 h), with the exception  
of LiN(CHMe2)2. Reactions with Et2O were somewhat slower.  
OSC.G 14 THERE ARE 14 CAPLUS RECORDS THAT CITE THIS RECORD (14 CITINGS)

L3 ANSWER 36 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1986:167908 CAPLUS

DN 104:167908

OREF 104:26579a,26582a

TI Low-temperature EPR and near-infrared MCD studies of highly anisotropic low-spin ferriheme complexes

AU Gadsby, Paul M. A.; Thomson, Andrew J.

CS Sch. Chem. Sci., Univ. East Anglia, Norwich, NR4 7TJ, UK

SO FEBS Letters (1986), 197(1-2), 253-7

CODEN: FEBLAL; ISSN: 0014-5793

DT Journal

LA English

AB Low-temperature ESR spectra and near-IR MCD spectra of the bis complexes of imidazole, 1-methylimidazole, 4-methylimidazole, 1,2-dimethylimidazole, and 2-methylimidazole with Fe(III) octaethylporphyrin (OEP) in the mixed organic solvent dichloromethane/diethyl ether are reported. The latter 2 complexes have highly anisotropic ESR spectra characteristic of the low-spin Fe(III) state. The optical charge-transfer bands have an unusually high MCD intensity, with a narrow linewidth. This feature may be typical of bis-histidine-ligated heme in cytochromes with sterically strained coordination and may be used to diagnostic such a conformation. The ESR and near-IR MCD spectra of the bis-butylamine complex of Fe(III) OEP are also reported. Bis-amine-ligated heme may be clearly distinguished from sterically hindered bis-imidazole (bis-histidine) by near-IR MCD spectroscopy, whereas the assignment is uncertain using ESR spectroscopy alone.

OSC.G 5 THERE ARE 5 CAPLUS RECORDS THAT CITE THIS RECORD (5 CITINGS)

L3 ANSWER 37 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1985:148717 CAPLUS

DN 102:148717

OREF 102:23353a,23356a

TI Secondary aminoether alcohols

IN Stogryn, Eugene L.; Ho, W. S. Winston; Montagna, Angelo A.; Sartori, Guido

PA Exxon Research and Engineering Co., USA

SO U.S., 9 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4487967	A	19841211	US 1983-565097	19831223
	CA 1228368	A1	19871020	CA 1984-465354	19841012
	NO 8404388	A	19850624	NO 1984-4388	19841105
	NO 160362	B	19890102		
	NO 160362	C	19890412		
	EP 147990	A1	19850710	EP 1984-308791	19841217
	EP 147990	B1	19880622		
	R: BE, CH, DE, FR, GB, IT, LI, NL				
	AU 8437096	A	19850704	AU 1984-37096	19841221
	AU 568708	B2	19880107		
	JP 60169450	A	19850902	JP 1984-270550	19841221
	JP 04081978	B	19921225		
	BR 8406684	A	19851022	BR 1984-6684	19841221
FRAI	US 1983-565097	A	19831223		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB A severely sterically hindered secondary amino ether alc. was prepared from a primary amine and a polyalkenyl ether glycol over a supported hydrogenation catalyst at elevated temps. Thus, Ni-Cu-Co/SiO<sub>2</sub>/kieselguhr (Trimetallic 2330) activated with H<sub>2</sub> at 350° catalyzed the reaction of Me<sub>3</sub>CNH<sub>2</sub> with (HOCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>O (2:1) to give 40% Me<sub>3</sub>CNH(CH<sub>2</sub>)<sub>2</sub>O(CH<sub>2</sub>)<sub>2</sub>OH (I) and byproduct N-tert-butylmorpholine (II) (I/II = 4.3). Also used as catalyst in this reaction were supported Ni, supported Rh, etc.

OSC.G 10 THERE ARE 10 CAPLUS RECORDS THAT CITE THIS RECORD (10 CITINGS)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 38 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1983:73244 CAPLUS

DN 98:73244

OREF 98:11227a,11230a

TI Kinetics of a sterically hindered amine-cured epoxy resin system

AU Buckley, Leonard J.; Roylance, David K.

CS Nav. Air Dev. Cent., Warminster, PA, USA

SO SAMPE Quarterly (1982), 14(1), 8-13

CODEN: SAMQA2; ISSN: 0036-0821

DT Journal

LA English

AB Fourier-transform IR spectroscopy (FT-IR) and torsional braid anal. (TBA) were used to study the reaction of an epoxy resin system cured with a sterically hindered amine. The system consisted of a 2/3:1/3 stoichiometric mixture of DER 332 [25085-99-8] and o-cresol-formaldehyde novolak resin polyglycidyl ether (ECN 1299) hardened with 2,5-dimethyl-2,5-hexanediamine [23578-35-0]. TBA showed the apparent activation energy to be .apprx.42 kJ/mol. The reaction kinetics was also considered from the unreacted freshly mixed condition and a partially reacted B-staged condition using IR spectroscopy. The B-staged condition was unreactive and stable at room temperature due to the quenching of the primary amine reaction by the glassy structure and the steric hindrance of the secondary amine reaction. Apparent activation energies for these 2 conditions were 4.94 kJ/mol and 49.0 kJ/mol, resp. The storage life at room temperature of the B-staged resin system was predicted to be  $\geq 3$  mo based on extrapolation of the exptl. kinetic data.

L3 ANSWER 39 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1981:572712 CAPLUS

DN 95:172712

OREF 95:28841a,28844a

TI Compositions for stabilizing electrolytes in lithium/titanium disulfide systems

IN Rao, Bhaskara M. L.; Eustace, Daniel J.; Farcasiu, Dan

PA Exxon Research and Engineering Co. , USA

SO U.S., 6 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	US 4284692	A	19810818	US 1980-144680	19800428
PRAI	US 1980-144680		19800428		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB A Li-TiS<sub>2</sub> battery has an electrolyte of  $\geq 1$  Li salt (LiAsF<sub>6</sub>) a cyclic ether (dioxolane [646-06-0]-MeOCH<sub>2</sub>CH<sub>2</sub>OMe) solvent, and an inhibitor of the polymerization of the cyclic ether by TiS<sub>2</sub>. The inhibitor is selected from LixTiS<sub>2</sub> (x .apprx.0.025-0.1) and/or  $\geq 1$  sterically hindered amine which does not intercalate with TiS<sub>2</sub>. The possible amines are:  
 1,8-bis(dimethylamino)naphthalene [20734-58-1];  
 2,2,6,6-tetramethylpiperidine; a polymeric pyridine such as poly(vinylpyridine) or 2-vinylpyridine-styrene copolymers [24980-54-9];  
 or tetramethylenediamine.

OSC.G 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)



L3 ANSWER 40 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1981:498884 CAPLUS

DN 95:98884

OREF 95:16627a,16630a

TI Stabilization of polyether polyols and polyurethane foams prepared therefrom

IN Hinze, Kenneth J.

PA Dow Chemical Co., USA

SO U.S., 5 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	US 4275173	A	19810623	US 1980-141854	19800421
PRAI	US 1980-141854		19800421		

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT

AB Polyurethane foams are prepared from polyols stabilized against oxidative degradation with a synergistic combination of sterically hindered phenols, 4,4'-bis( $\alpha$ , $\alpha$ -dimethylbenzyl)diphenylamine (I) [10081-67-1], and phenothiazine (II) [92-84-2]. Thus, a scorch-resistant polyurethane [57516-88-8] foam was prepared from a polyethylenepolypropylene glycol glycerol ether [9082-00-2] (containing 2,6-di-tert-butyl-4-methylphenol [128-37-0] 1900, I 1000 and II 100 ppm) 200, water 10.4, L-540 silicone surfactant 2.4, Niax A-6 amine catalyst 0.25, T-9 stannous octoate 0.4 and TDI 141.9 g.

OSC.G 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

L3 ANSWER 41 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1980:533555 CAPLUS

DN 93:133555

OREF 93:21303a,21306a

TI Polyethylene compositions for electric insulators

PA Dainichi Nippon Cables, Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 3 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 55065245	A	19800516	JP 1978-138996	19781110
PRAI	JP 1978-138996	A	19781110		
AB	Polyethylene (I) [9002-88-4] elec. insulator comps. contain sterically hindered phenol (and/or S-containing ether) and amine antioxidants. For example, a dicumyl peroxide-cured I specimen containing 0.3 phr 4,4'-thiobis(6-tert-butyl-3-methylphenol) (II) [96-69-5] and 0.2 phr 4,4'-bis( $\alpha,\alpha$ -dimethylbenzyl)diphenylamine (III) [10081-67-1] had better treeing resistance (90° water) than a control containing 0.5 phr II and no III.				

L3 ANSWER 42 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1977:189100 CAPLUS  
 DN 86:189100  
 OREF 86:29653a,29656a  
 TI Oxonium salt alkylation of structurally and optically labile alcohols  
 AU Diem, Mary Jo; Burow, D. F.; Fry, James L.  
 CS Bowman-Oddy Lab., Univ. Toledo, Toledo, OH, USA  
 SO Journal of Organic Chemistry (1977), 42(10), 1801-2  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DT Journal  
 LA English  
 AB The acid-sensitive alcs. Me3CCH2OH, (R)-(+)-PhCHMeOH, and  
 (S)-(-)-EtCHMeCH2OH were converted to their Et ethers by treatment with  
 Et3O+ BF4- in CH2Cl2 in the presence of (iso-Pr)2NEt (I). O-methylation  
 using Me3O+ BF4- was carried out in the presence of the less nucleophilic  
 base 1,8-bis(dimethylamino)naphthalene, since I was rapidly N-methylated.  
 Treatment of 2-tert-butyl-2-adamantanol with the above oxonium salt-  
 sterically hindered amine reagents did not  
 give the Me or Et ether.  
 OSC.G 23 THERE ARE 23 CAPLUS RECORDS THAT CITE THIS RECORD (23 CITINGS)

L3 ANSWER 43 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1967:464543 CAPLUS  
 DN 67:64543  
 OREF 67:12179a,12182a  
 TI Preparation of aniline chromium tricarbonyl compounds  
 IN Whiting, Mark C.  
 PA Ethyl Corp.  
 SO U.S., 5 pp.  
 CODEN: USXXAM  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3317522		19670502	US 1963-264025	19630311
PRAI	GB		19620312		

GI For diagram(s), see printed CA Issue.

AB Aniline Cr tricarbonyl compds. are synthesized by substitution of F in Ia, where X is H or alkyl, by the corresponding amine R<sub>1</sub>R<sub>2</sub>NH (R<sub>1</sub> and R<sub>2</sub> are H, alkyl or aryl groups). Slow reactions with sterically hindered amines can be accelerated with catalytic quantities of a non-alc. polar solvent (acetamide, formamide, dimethylformamide, acetonitrile, and Me<sub>2</sub>SO). Thus, HMeNPhCr(CO)<sub>3</sub> (I) is obtained in 84% yield by treating 92.7 g. FPhCr(CO)<sub>3</sub> and 270 g. MeNH<sub>2</sub> for 12 min. at 21°. I m. 123.5° (petroleum ether). Similarly prepared were Ia (X = H) (amine used, % yield, and m.p. given: Me<sub>2</sub>NH, 84, 144.5-45°; hexylamine, 80, 67-8°; cyclohexylamine, 82, 132-3°; isopropylamine, 85, 102-3°; piperidine, 94, 125-6.5°; pyrrolidine, 92, 161-2°; morpholine, 85, 171-2.5°; benzylamine, 87, 129-30°; Et<sub>2</sub>NH, 70, 113-15°; EtNH<sub>2</sub>, 11, 112-16°; tert-BuNH<sub>2</sub>, 88, 126-8°. Ia are useful as petroleum additives, chemical intermediates, fungicides, herbicides, bactericides, and pesticides.

OSC.G 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

L3 ANSWER 44 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN  
 AN 1965:15204 CAPLUS  
 DN 62:15204  
 OREF 62:2738g-h,2739a-c  
 TI Mono and diaryl phosphites  
 IN Shepard, Alvin F.; Dannels, Bobby F.; Kujawa, Francis M.  
 PA Hooker Chemical Corp.  
 SO 20 pp.  
 DT Patent  
 LA French  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	FR 1366579		19640710	FR 1963-945453	19620831
	BE 651633			BE	
	NL 297350			NL	
	US 3271481		19660906	US 1962-220902	19620831
PRAI	FR		19620831		

AB The title compds. were prepared from sterically hindered phenols and PC13 with removal of the HCl formed. The products could be used as stabilizers, plasticizers, fire-retardants, or lubricant additives. To a mixture of 463 g. PC13 and 121.4 g. NEt3, 265 g. of 2,4,6-tri-tert-butylphenol was added slowly at 10° under a N atmospheric, the mixture refluxed 6 hrs., Et3N.HCl filtered off and washed with petr. ether, and the combined filtrates distilled to give a fraction, b0.5 119-24°. On chilling, 313 g. of a white crystalline solid was obtained. After grinding, this 2,4,6-tri-tert-butylphenyl dichlorophosphite was kept 2 hrs. with 4 l. H2O, filtered off, dried, and recrystd. (dimethoxyethane), m. 185°. In one preparation, the Na salt of 2,6-di-tert-butylphenol was used in the absence of a tertiary amine. Also, CaO or a vacuum were used in similar preps. Also prepared were 2,6-di-tert-butylphenyl dichlorophosphite, pale yellow, b1 143-6°; 2,6-ditert-butyl-4-methylphenyl dichlorophosphite, b0.5 117-20° (solidified on standing); 2,6-di-tert-butyl-4-nonylphenyl dichlorophosphite, b2.5 148-154°; 2,6-di-tert-butyl-4-chlorophenyl dichlorophosphite, b2.6 156-66°; 2,4,6-tris-(phenethyl)phenyl dichlorophosphite; mono(2,6-di-tert-butylphenyl) phosphite, m. 136.5-38° (hexane); mono(2,6-di-tert-butyl-4-methylphenyl) phosphite, m. 190-5°; mono(2,6-di-tert-butyl-4-nonylphenyl) phosphite, a viscous oil (Na salt had good surface active properties); mono(2,6-di-tert-butyl-4-chlorophenyl) phosphite, m. 140-2° (dimethoxyethane); and mono[2,4,6-tris(phenethyl)phenyl] phosphite, a viscous oil. Diaryl chlorophosphites were prepared as follows. A mixture of 112 g. di-tert-butyl-4-methylphenol and 117 g. NEt3 was heated to 75°. After a clear solution formed, 20.5 g. PC13 was added over 1.5 hrs., the mixture heated 19 hrs. at 111-13°, cooled, 156 g. petr. ether added, the solids washed with petr. ether, the combined filtrates distilled in vacuo to eliminate excess phenol as well as solvent, the residue (58 g.) ground and treated with 11.5% aqueous NaOH, and the solid filtered off, washed with H2O, and recrystd. from Me2CO to give 2,6-di-tert-butyl-4-methylphenyl monochlorophosphite, m. 113-15°. This (10 g.) was hydrolyzed in 500 cc. 0.1N HCl by refluxing 24 hrs. in an inert atmospheric. After filtration, washing free of Cl-, and drying, the bis(2,6-di-tert-butyl-4-methylphenyl) phosphite m. 161-3°. Recrystn. from hexane did not alter the m.p. appreciably. Also prepared were bis(2,6-di-tert-butyl-4-chlorophenyl) chlorophosphite, b2.6 156-66°; bis(2,4,6-tri-tert-butylphenyl) chlorophosphite, m. 173-4° (hexane); bis(2,6-di-tert-butylphenyl) chlorophosphite; bis(2,6-di-tert-butyl-4-chlorophenyl) phosphite, m. 147-8.5°; bis(2,6-di-tert-butylphenyl) phosphite, m. 147-9° (Me2CO-hexane);

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and (2,6-di-tert-butylphenyl) diphenyl phosphite, b. 200-2°. The products were quite stable to hydrolysis.

L3 ANSWER 45 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1964:45745 CAPLUS

DN 60:45745

OREF 60:8035f-h,8036a-d

TI Synthesis and cyclodimerization of stable  
1-tert-alkylamino-2,3-epoxypropanes-related sterically promoted  
eight-membered ring closures

AU Gaertner, V. R.

CS Monsanto Chem. Co., St. Louis, MO

SO Tetrahedron Letters (1964), (3-4), 141-6

CODEN: TELEAY; ISSN: 0040-4039

DT Journal

LA Unavailable

OS CASREACT 60:45745

GI For diagram(s), see printed CA Issue.

AB Exceptionally stable secondary amino epoxides, their cyclodimerization to diazacyclooctanediols, and novel 8-membered ring closures are described. Equimolar reaction of tert-alkylamines with epichlorohydrin (I), either reactant being present in 25-100% excess in MeOH at 20-5° followed by dehydrohalogenation with 40-50% aqueous alkali below 35° of the crude 1-tert-alkylamino-3-chloro-2-propanols gave the following 1-tert-alkylamino-2,3-epoxypropanes (II) (alkyl group, b.p./mm., n<sub>D</sub><sup>25</sup>, and % yield given): tert-Bu (III), 54-5°/10, 1.4307, 58; tert-BuCH<sub>2</sub>CM<sub>2</sub> (IV), 65-6°/1, 1.4492, 66. When atmospheric moisture was excluded, pure III and IV could be kept at room temperature for some time without measurable change. H<sub>2</sub>O or alcs. catalyzed the disappearance of the oxirane ring. In MeOH at 25.0°, the 2nd order constant for the initial dimerization of III was 7.0 + 10<sup>-6</sup> l. mole<sup>-1</sup> sec.<sup>-1</sup> This rate is only 3.5-fold slower than the initial reaction of the less hindered tert-BuNH<sub>2</sub> with I (k<sub>2</sub> = 2.5 + 10<sup>-5</sup> l. mole<sup>-1</sup> sec.<sup>-1</sup> at 25.0° in 96% MeOH). The stability of II was attributable more to the absence of H-bonding catalysts than to the steric effect. After 2 months in MeOH at 20-5°, less than 10% of the original weight of III was not volatile up to 220°/1 mm. Crystallization of the distillate from heptane and then from petr. ether gave 2 major products, V (R = R<sub>1</sub> = tert-Bu) (VI), one (23% yield) m. 124-5°, and the other m. 69-72° (incompletely separated from unidentified oils). Three alternate syntheses of V were mentioned. The different behaviors of the OH nuclear magnetic resonance peaks upon dilution suggested that the higher and lower melting diols were trans and cis isomers, resp. Another example of cyclodimerization of 1-amino-2,3-epoxypropanes indicated that the reaction was not limited to highly hindered starting materials. 1-Anilino-2,3-epoxypropane (VII) gave 17% V (R = R<sub>1</sub> = Ph) (VIII), m. 212-13° also obtained (25% yield) from PhNH<sub>2</sub> and N,N-bis(2,3-epoxypropyl)aniline (IX). An equimolar mixture of III and VII gave VI, VIII, and a trace V (R = tert-Bu, R<sub>1</sub> = Ph) (X), m. 158-9°. The reaction of a primary amine with a diglycidylamine was also general and provided V carrying 2 different substituents. Either PhNH<sub>2</sub> and tert-butylbis(2,3-epoxypropyl)amine (XI) or tert-BuNH<sub>2</sub> and IX gave X in 28 and 24% yields, resp. From the 2 appropriate pairs was similarly prepared V (R = Bu, R<sub>1</sub> = Ph), m. 133-4°. In these cases only 1 pure crystalline compound, probably the trans isomer, was isolated. A similar 8-membered ring closure, involving an intermediate mercaptide, occurred in the rapid exothermic reaction of Na<sub>2</sub>S with XI to give 27% 1-tert-butyl-1-aza-5-thia-3,7-cyclooctanediol, m. 94.5-5.0°. The structure was established by hydrogenolysis to tert-BuN(CH<sub>2</sub>CHMeOH)<sub>2</sub> (XII) (isolated by vapor phase chromatography), identical (infrared spectrum) with XII prepared from tert-BuNH<sub>2</sub> with excess propylene oxide (63% yield, b<sub>0.6</sub> 85°, n<sub>D</sub><sup>25</sup> 1.4558). These cyclizations are rationalized by a sterically favored conformer of solvated intermediate of type XIII

10/591,778

(A = NHR or S-) which both promotes ring formation and inhibits  
polymerization.

OSC.G 2 THERE ARE 2 CAPLUS RECORDS THAT CITE THIS RECORD (2 CITINGS)



L3 ANSWER 46 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1961:143790 CAPLUS

DN 55:143790

OREF 55:27155e-i,27156a-i

TI New class of local anesthetics. Hydroxyalkyliminobisacetamides

AU Freed, Meier E.; Bruce, William F.; Hanslick, Roy S.; Maschitti, Albert

CS Wyeth Labs., Philadelphia, PA

SO Journal of Organic Chemistry (1961), 26, 2378-83

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

AB cf. CA 53, 6088e. A series of hydroxyalkyliminobisacetamides, HOXN(CH<sub>2</sub>CONRR1)CH<sub>2</sub>CONR2R3 (where X is alkylene, cycloalkylene, or aralkylene, R, R1, R2, R3 represent lower alkyl or aralkyl, and where RR1 may or may not equal R2R3), were prepared, examined for local anesthetic action, and studied for structure-activity relationships. The preparation of all chloroacetamides, hydroxyalkylaminoacetamides, hydroxyalkyliminoacetamides and their esters were carried out essentially in the same manner. PhCH<sub>2</sub>CMe<sub>2</sub>NHMe (0.86 mole) in 500 ml. PhMe stirred 1 hr. at -15° with addition of 0.40 mole ClCH<sub>2</sub>COCl, the mixture filtered at 20°, the amine HCl salt washed with PhMe, the combined filtrate and washings dried, and the residue on evaporation distilled yielded 70.5% PhCH<sub>2</sub>CMe<sub>2</sub>NMeCOCH<sub>2</sub>Cl (I), b<sub>0.5</sub> 140-1°. HOCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> (0.1 mole) and 30 g. anhydrous powdered Na<sub>2</sub>CO<sub>3</sub> in 300 ml. well-stirred boiling BuOH slowly treated with 0.1 mole I in 50 ml. BuOH, the mixture refluxed 12 hrs., cooled, and filtered, and the residue on evaporation crystallized from C<sub>6</sub>H<sub>14</sub> yielded

63% HOCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CONMeCMe<sub>2</sub>CH<sub>2</sub>Ph, m. 74.5-6.5°; HCl salt, m.

163-4°. Similarly were prepared and tabulated

hydroxyalkylaminoacetamides, RNHCH<sub>2</sub>CONR1Me (R, R1, and m.p. HCl salt

given): PhCHOHCH<sub>2</sub>, PhCH<sub>2</sub>CMe<sub>2</sub>, 201-2°; PhCHOHCH<sub>2</sub>Me<sub>2</sub>, PhCH<sub>2</sub>CMe<sub>2</sub>,

189-90°; HOCH<sub>2</sub>CH<sub>2</sub>CMe<sub>2</sub>, PhCH<sub>2</sub>CMe<sub>2</sub>, 169-70°; (HOCH<sub>2</sub>)<sub>2</sub>CH,

PhCH<sub>2</sub>CMe<sub>2</sub>, 175-6°; HOCHMeCH<sub>2</sub>, PhCH<sub>2</sub>, 134-5°. I (0.1 mole)

and 20 g. K<sub>2</sub>CO<sub>3</sub> in 250 ml. boiling BuOH stirred with addition of 0.05 mole

freshly distilled HOCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, the mixture refluxed 20 hrs. and the cooled

mixture filtered, the filtrate washed (aqueous 5% Na<sub>2</sub>CO<sub>3</sub>, H<sub>2</sub>O) and the dried (MgSO<sub>4</sub>) solution evaporated in vacuo yielded 71% hydroxyalkyliminobisacetamide, RN(CH<sub>2</sub>CONR1R2)<sub>2</sub> (II) (R = HOCH<sub>2</sub>CH<sub>2</sub>, R1 = Me, R2 = PhCH<sub>2</sub>CMe<sub>2</sub>) (III), m.

104-5°; HCl salt m. 146-7° (MeOH-Me<sub>2</sub>CO); nicotinic acid

ester m. 158-9°. III (20 g.) in 100 ml. dry CHCl<sub>3</sub> treated with 5

g. SOCl<sub>2</sub> in 25 ml. CHCl<sub>3</sub>, the mixture stirred 3 hrs., and the residue on

evaporation crystallized from alc.-Et<sub>2</sub>O yielded 79 g. II (R = ClCH<sub>2</sub>CH<sub>2</sub>, R1 =

Me, R2

= PhCH<sub>2</sub>CMe<sub>2</sub>) HCl salt (IV), m. 155-6° (alc.-Et<sub>2</sub>O). IV (3 g.) in 20

ml. MeOH containing 3 g. anhydrous NH<sub>3</sub> heated 18 hrs. at 90° in a pressure

tube, the cooled mixture and MeOH rinsings filtered from NH<sub>4</sub>Cl, freed from

MeOH and excess NH<sub>3</sub>, and taken up in 50 ml. Me<sub>2</sub>CHOH, and the filtered

solution treated with dry HCl and diluted with 150 ml. dry Et<sub>2</sub>O yielded 40.5%

II (R = H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>, R1 = Me, R2 = PhCH<sub>2</sub>CMe<sub>2</sub>), m. 231-2°. III (0.02

mole) in 150 ml. dry Et<sub>2</sub>O added slowly with stirring to 1.8 g. LiAlH<sub>4</sub> in

300 ml. dry Et<sub>2</sub>O, the mixture refluxed 25 hrs. before cautious decomposition

with

8 ml. H<sub>2</sub>O, the dried Et<sub>2</sub>O layer treated with HCl, the oily product

trituated with Me<sub>2</sub>CO, and the product (29.3%) recrystd. from MeOH/Me<sub>2</sub>CO

yielded HOCH<sub>2</sub>CH<sub>2</sub>N(CH<sub>2</sub>CH<sub>2</sub>NMeCMe<sub>2</sub>CH<sub>2</sub>Ph)<sub>2</sub>, m. 229-30° (decomposition);

tri-HCl salt m. 239-40°; MeI salt, m. 122-3°; tri-MeI salt,

m. 154-5°. To obtain the bis compds. with sterically

hindered amino alcs., the use of a higher boiling solvent (such as

PhOMe) was necessary. Phys. and pharmacol. data are tabulated for the

various series of compds., RN(CH<sub>2</sub>CONR1R2)<sub>2</sub> (R, R1, R2, b.p./mm., duration

of activity on rabbit cornea and % solution given): HOCH<sub>2</sub>CH<sub>2</sub>, (R<sub>1</sub>R<sub>2</sub>=)CH<sub>2</sub>CH<sub>2</sub>, 203-5°/1.0, neg., 0.1; HOCH<sub>2</sub>CH<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 208-10°/0.5, 25 min., 0.01; MeCHOHCH<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 200-5°/0.1, neg., 0.1; HOCH<sub>2</sub>CH<sub>2</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, 170-1°/0.5, neg., 0.1; HO(CH<sub>2</sub>)<sub>3</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, 190-2°/0.5, neg., 0.1; HOCH<sub>2</sub>CMe<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 155-60°/0.5, neg., 0.1; HOCH<sub>2</sub>CMe<sub>2</sub>, MeCH<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 170-5°/0.5, neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>4</sub>, Me(CH<sub>2</sub>)<sub>4</sub>, 230-5°/1.0, neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, C<sub>6</sub>H<sub>11</sub>, C<sub>6</sub>H<sub>11</sub>, - (HCl salt m. 215-16°), neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>5</sub>, Me(CH<sub>2</sub>)<sub>5</sub>, 194-6°/0.5, 48 min., 0.1. For RN(CH<sub>2</sub>CONR<sub>1</sub>R<sub>2</sub>)<sub>2</sub> [R, R<sub>1</sub>, R<sub>2</sub>, m.p. of base or HCl salt (or b.p./mm.), duration in min. and % solution given): HO(CH<sub>2</sub>)<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 104-4.5°, 25, 0.0005; HOCHMeCH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 113-14°, 28, 0.0001; HOCH<sub>2</sub>CH<sub>2</sub>Et, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 144-5° (HCl salt), 37, 0.0005; HO(CH<sub>2</sub>)<sub>3</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 164-5° (HCl salt), 82, 0.1; HO(CH<sub>2</sub>)<sub>6</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 250-60°/0.002, neg., 0.1; (HOCH<sub>2</sub>)<sub>3</sub>C, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 157-8°, 24, 0.001; 2-HOC<sub>6</sub>H<sub>10</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 108.0-8.5°, 75, 0.0025; PhCHOHCH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 182-3° (HCl salt), 24, 0.001; PhCHOHCH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 203-4° (HCl salt), neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me, C<sub>6</sub>H<sub>11</sub>, 190-5°/1.0, neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, H, PhCH<sub>2</sub>CH<sub>2</sub>, 72-3° (HCl salt), neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, PhCH<sub>2</sub>, 118° (HCl salt), 29, 0.001; HOCHMeCH<sub>2</sub>, Me(CH<sub>2</sub>)<sub>5</sub>, PhCH<sub>2</sub>, 195-200°/0.05, 44, 0.1; HOCHMeCH<sub>2</sub>, H, 2,6-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 193-4° (HCl salt), neg. 0.1. For RN(CH<sub>2</sub>CONR<sub>1</sub>R<sub>2</sub>)CH<sub>2</sub>CONR<sub>3</sub>R<sub>4</sub> (R, R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, R<sub>4</sub>, b.p./mm. or m.p. of base or HCl salt, duration, and % solution given): HO(CH<sub>2</sub>)<sub>2</sub>, MeCH<sub>2</sub>, MeCH<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 203-5°/1.0, 29, 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, 198-200°/0.5, 21, 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, MeCH<sub>2</sub>, MeCH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 121-2°, neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me(CH<sub>2</sub>)<sub>4</sub>, Me(CH<sub>2</sub>)<sub>4</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 92-3°, 63, 0.1; HO(CH<sub>2</sub>)<sub>3</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, Me<sub>2</sub>CHCH<sub>2</sub>, Me, C<sub>6</sub>H<sub>11</sub>, 205-80°/1.0, neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, Me, PhCH<sub>2</sub>CHMe, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, hygroscopic, 55, 0.001; HO(CH<sub>2</sub>)<sub>2</sub>, H, PhCH<sub>2</sub>CH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 158° (HCl salt), neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, H, HOCH<sub>2</sub>CH<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 42° (HCl salt), neg., 0.1; HO(CH<sub>2</sub>)<sub>2</sub>, H, Me(CH<sub>2</sub>)<sub>5</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 260°/1.0, 9, 0.05. For XCH<sub>2</sub>CH<sub>2</sub>N(CH<sub>2</sub>CONR<sub>1</sub>R<sub>2</sub>)<sub>2</sub> (X, R<sub>1</sub>, R<sub>2</sub>, m.p. HCl salt, duration, and % solution): MeCO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 169-70°, 32, 0.001; Me(CH<sub>2</sub>)<sub>10</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 143-5°, 38, 0.01; p-MeC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 168-9°, 42, 0.001; p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 168-9°, 27, 0.0005; MeCO<sub>2</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, Me(CH<sub>2</sub>)<sub>3</sub>, 212-14°/0.05(base), 32, 0.01; m-ClC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 87-8° (base, from Me<sub>2</sub>CHOH-petr. ether), active, 0.1; (3-C<sub>5</sub>H<sub>4</sub>N)CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 158-9°, 35, 0.0005; p-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 126-7°, active, 0.1; p-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>, Me, PhCH<sub>2</sub>CMe<sub>2</sub>, 199-200°, active, 0.1. Iminoacetamides in which the amido N was derived from aliphatic amines had relatively little local anesthetic action and were more toxic than those derived from aralkyl amines. The use of PhCH<sub>2</sub>CMe<sub>2</sub>NHMe produced the highest degree of local anesthetic activity in II. Substitution of PhCH<sub>2</sub>CHMeNHMe in 1 amide group halved the activity. In the alkanolamine moiety, use of a sterically hindered base (H<sub>2</sub>NMe<sub>2</sub>CH<sub>2</sub>OH) markedly reduced activity. Separation of HO from the tertiary amino group by interposition of CH<sub>2</sub> groups reduced activity. The activity of HOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>N(CH<sub>2</sub>CONMeCMe<sub>2</sub>CH<sub>2</sub>Ph)<sub>2</sub> was 1/500 of that of the homologous HOCH<sub>2</sub>CH<sub>2</sub>N(CH<sub>2</sub>CONMeCMe<sub>2</sub>CH<sub>2</sub>Ph)<sub>2</sub>. Replacement of HO by NH<sub>2</sub> or Cl, and quaternization of the tertiary amine or reduction of the amide groups to tertiary amines all resulted in nearly complete loss of activity. The activity was not increased by ester formation.

OSC.G 2 THERE ARE 2 CAPLUS RECORDS THAT CITE THIS RECORD (2 CITINGS)

L3 ANSWER 47 OF 48 CAPLUS COPYRIGHT 2010 ACS on STN

AN 1955:19886 CAPLUS

DN 49:19886

OREF 49:3870h-i,3871a-i,3872a-i,3873a-b

TI Organic lithium derivatives

AU Thiec, Jeanne

SO Ann. chim. (Paris) (1954), 9, 51-96

DT Journal

LA Unavailable

AB The carbonation of PhLi (I), o-MeC6H4Li (II), 2,4,6-(Me2CH)3C6H2Li (III), 2,3,5,6-(Me2CH)4C6HLi (IV), 2,5-(Me3C)2C6H3Li (V), and bornyllithium (VI) (containing about 20% isobornyl-lithium) from pinene-HCl (VII) has been studied by 2 methods. The oxidation of VI with O and the reaction of VI with various ketones were also investigated. Method A for the carbonation of the organo-Li compds. consisted in passing a stream of dry CO2 over the surface of the Li derivative in dry Et2O; in method B the solution of the Li derivative was introduced through a nozzle into an atmospheric of CO2. I

prepared from

1/6 mol PhBr in Et2O (1 mol/1300 cc.) gave by method A at 17-30° 58% PhBz and no BzOH. I (0.1443 mol) in Et2O (1 mol/4900 cc.) yielded by method B at 0° 72% BzOH and 8% Ph3COH (VIII); 1 mol I in 1300 cc. Et2O at 0° yielded similarly 31% BzOH and 33% VIII; and 1 mol I in 4900 cc. Et2O at 16-19° gave 32% BzOH and 35% VIII. The reaction by method A can be expressed by the equations: 2I + CO2 → Ph2C(OLi)2 (IX); IX + CO2 → BzPh + Li2CO3; whereas by method B in the presence of an excess CO2 at all times, the reaction seems to proceed according to the equations: I + CO2 → BzOLi; BzOLi + I + CO2 → BzPh + Li2CO3. II (1 mol) in 1300 cc. Et2O gave by method A at room temperature 30% o-MeC6H4CO2H (X); in 4900 cc. Et2O by method B the yield

of

X was 69%. 2,4,6-(Me2CH)3C6H2Br (XI), b18 146-8°, mixed with 20% PhBr to initiate the reaction, was converted to 50-73% III (in 1 run 90% was obtained). III, prepared from 1/15 mol XI and 1/75 mol PhBr in Et2O (1 mol/1300 cc. Et2O) carbonated by method A at 17-25° yielded 53% 2,4,6-(Me2CH)3C6H2CO2H (XII), dark yellow solid, m. 178-80°, and 32% recovered XI. III, prepared from 1/12 mol XI and 1/60 mol PhBr in Et2O (1 mol/4900 cc.) gave by method B at 0° 87% XII, white solid, m. 185-7°, and 15% recovered XI, and at 14-16° in a similar run 76% XII, m. 176-9°. The steric effect in the carbonation of III leads to the formation of 53% XII by method A, whereas I under the same conditions did not give BzOH. Since no BzOH was formed in the runs with III from the added PhBr, the formation of the III proceeds apparently by the reaction of XI with I. 1,2,4,5-C6H2(CHMe2)4 (XIII), m. 118°, was prepared in 65% yield from com. iso-PrOH and C6H6 in the presence of 20% oleum by the method of Kirmann and Graves (C.A. 29, 2516.8). XIII treated in CHCl3 in the cold with Br without a catalyst and the mixture distilled gave at 160-80°/20mm. 40% 2,3,5,6-(Me2CH)4C6HBr (XIV), needles, m. 148-9°. Li did not react with XIV in Et2O or petr. ether. XIV (0.03 mol) added to 0.075 mol BuLi in petr. ether (1 mol/2600 cc.), and the mixture refluxed 5 h. and carbonated 24 h. by method A at 17° gave 4.26 g. (52%) 2,3,5,6-(Me2CH)4C6HCO2H (XV) and 30% mixture of XIII and XIV, m. 100°. XV sublimed at about 100° and melted about 240° (hot Maquenne block). p-C6H4(CMe3)2 (XVI), m. 75-6°, was prepared from Me2CHCH2OH and C6H6 in the presence of 30% oleum or 80% H2SO4. XVI in CCl4 treated with an equivalent amount of Br in the presence of Fe gave 56% recovered XVI and 27% 2,5-(Me3C)2C6H3Br (XVII). XVI treated similarly with 1.75 mol equivs. Br gave a colorless liquid product, b18 154-6°, consisting of 82% XVII and 18% di-Br derivative which was used for the preparation of V. BuLi (1.5

mol)

in Et<sub>2</sub>O treated with 1 mol crude XVII in Et<sub>2</sub>O and the mixture refluxed 0.5 h. and analyzed showed 92% organo-Li derivs.; after 2.5 h., 87%; and after 5 h. only 48%. Crude XVII (12 g.) treated with 17.3/269 mol BuLi in 102 cc. Et<sub>2</sub>O, and the mixture refluxed after 0.5 h. and distilled gave 3.5 g. (57%) BuBr and 5 g. (59%) XVI, m. 72°. Crude XVII (1 mol) added to 1.5 mol BuLi in Et<sub>2</sub>O and the mixture refluxed 0.5 h. gave a solution containing 59% V.

XVII (0.05 mol) treated with 0.075 mol BuLi in 95 cc. Et<sub>2</sub>O and the mixture carbonated by method A at about 18° yielded 54% crude 2,5-(Me<sub>3</sub>C)2C6H3CO<sub>2</sub>H (90% yield based on a 59% yield of V), m. 100-10° (recrystd. from aqueous EtOH and then petr. ether, m. 120-3°; purified through the NH<sub>4</sub> or the NMe<sub>3</sub> salt, m. 128°). Pinene, [α]<sub>D</sub><sup>25</sup> -38.6°, [α]<sub>D</sub><sup>25</sup> -43.5°, was converted to VII, m. 120-3° (from EtOH), [α]<sub>D</sub><sup>25</sup> -34.7°, [α]<sub>D</sub><sup>25</sup> -38.8° (c 0.017, EtOH). For the preparation of VI, VII was distilled in a small amount of PhMe up to 110° to remove the EtOH of crystallization VII treated in petr. ether (b. 35-40°) under N in the presence of a crystal of iodine with Li gave 49-54% VI. VII and 10 mol % BuCl added in 1 portion to the Li in petr. ether yielded 75% VI. VI (0.051 mol) (52% yield from VII) in Et<sub>2</sub>O (1 mol/4900 cc.) carbonated by method B yielded 51% essentially pure camphane-carboxylic acid (XVIII), [α]<sub>D</sub><sup>25</sup> -9.6°, [α]<sub>D</sub><sup>25</sup> -10.2° (c 0.025, PhMe). The yield of XVIII in a similar run but by method A (1 mol VI/4 l. petr. ether) was 64%; the crude XVIII yielded 43% pure XVIII, [α]<sub>D</sub><sup>25</sup> -11.2°, [α]<sub>D</sub><sup>25</sup> -12.8° (c 0.025, PhMe). VI treated at room temperature with CO<sub>2</sub> under slight pressure (about 50 cc. H<sub>2</sub>O) yielded 35.5% XVIII, [α]<sub>D</sub><sup>25</sup> -1.8°, [α]<sub>D</sub><sup>25</sup> -2.4°, and 17% dibornyl ketone (XIX), b<sub>p</sub> 178-80°, n<sub>D</sub><sup>20</sup> 1.502, [α]<sub>D</sub><sup>25</sup> -33.7° (c 4%, EtOH). The partial carbonation of VI during 3 min. with pure CO<sub>2</sub> and with a CO<sub>2</sub>-N mixture gave in 4 runs XVIII with values of [α]<sub>D</sub><sup>25</sup> between -3.6 and 9.2°. The yield of XIX from VI carbonated only to the extent of 50% was 22%, along with 30% XVIII. VI treated with CH<sub>2</sub>:CHCH<sub>2</sub>Br and then carbonated gave XVIII with [α]<sub>D</sub><sup>25</sup> -10.2°, indicating that no preferential attack of the bromide on the bornyl derivative occurred as with the Grignard derivative of VII. The crude XVIII obtained in these runs, recrystd. from HCO<sub>2</sub>H, gave the pure acid as needles, m. 78-80° [α]<sub>D</sub><sup>25</sup> -7.5°; recrystd. from PhMe, m. 82-4°, [α]<sub>D</sub><sup>25</sup> -4°. The color reaction of Gilman and Schulze (C.A. 19, 2443) for organometallic derivs. with Michler's ketone was also very sensitive with VI, and in general with compds. containing the Li attached to C, whereas compds. with the CO<sub>2</sub>Li grouping did not give the color test. VI (0.1067 mol) added dropwise under N to 0.0534 mol HCO<sub>2</sub>Et in a little petr. ether, and the mixture refluxed 4 h., hydrolyzed, and distilled yielded 8.1 g. (50%) dibornylcarbinol, b<sub>p</sub> 210-20°, which, crystallized twice from C<sub>6</sub>H<sub>6</sub>-EtOH, gave white crystals, m. 154-60°, [α]<sub>D</sub><sup>25</sup> -32.5°, [α]<sub>D</sub><sup>25</sup> -34.5°, [α]<sub>D</sub><sup>25</sup> -53° (c 0.02, PhMe). VI (0.108 mol) in petr. ether (1 mol/3380 cc.) agitated under pure O absorbed 80% of the theor. value; the mixture hydrolyzed, the solvent removed, and the residue steam distilled yielded 7 g. product, m. 175-85°, [α]<sub>D</sub><sup>25</sup> -30.33°, [α]<sub>D</sub><sup>25</sup> -35.66°, [α]<sub>D</sub><sup>25</sup> -79.66°, [α]<sub>D</sub><sup>25</sup> -436/[α]<sub>D</sub><sup>25</sup> 578 2.626, consisting of 35% borneol (XX), 22% isoborneol (XXI), and 43% camphor (XXII) VI (0.124 mol) in petr. ether (1 mol/3400 cc.) agitated at 0° under O without previous removal of the N from the apparatus gave 47% product, m. 192-202°, [α]<sub>D</sub><sup>25</sup> -19.0°, [α]<sub>D</sub><sup>25</sup> -21.3°, [α]<sub>D</sub><sup>25</sup> -41.66°, [α]<sub>D</sub><sup>25</sup> -436/[α]<sub>D</sub><sup>25</sup> 578 2.19 consisting of 56% XX, 30% XXI, and 6% XXII, which, recrystd. from petr. ether, yielded a mixture, m. 198-203°, of 69% XX, 26% XXI, and 5% XXII. VI (0.1268 mol) stirred

at 0° under a low partial O pressure, the apparatus flushed with O, and the mixture further oxidized at room temperature absorbed 58 and 75% O, resp., and

gave 11.5 g. (60%) mixture, m. 175-87°,  $[\alpha]_D^{25}$  -28.66°,  $[\alpha]_D^{25}$  F46 -34.0°,  $[\alpha]_D^{25}$  436 -75.0°,  $[\alpha]_D^{25}$  436/ $[\alpha]_D^{25}$  578 2.61, consisting of 37% XX, 23% XXI, and 40% XXII. On the basis of these expts. the VI appears to be a mixture of 70% bornyl-lithium and 30% isobornyl-lithium, as compared to the 80:20% ratio determined by the carbonation expts. Me3CCOCl treated with PhMgBr gave 62% Me3CBz, b17 112-14° nD20 1.5103. 1,3,5-C6H3(CHMe2)3 (1 mol) in glacial AcOH treated with 1.5 mol HNO3 gave 95.5% 2,4,6-(Me2CH)3C6H2NO2 (XXXIII), m. 72-3°. XXXIII (0.1 mol) hydrogenated in 250 cc. iso-PrOH over 40 g. Raney Ni 2 h. at room temperature gave 75% 2,4,6-(Me2CH)3C6H2NH2 (XXIV), b18 158-60°, nD20 1.5211; HCl salt, white powder, m. 245-7°, insol. in H2O. XXIV (75.2 g.) treated with 225 g. MeI and 170 g. Na2CO3 in 1125 cc. H2O heated several hrs. on the water bath, the mixture cooled, and the solid product dissolved in Et2O treated with KOH, and recrystd. gave 81% crude N,N-di-Me derivative (XXV) of XXIV, yellowish crystals, m. 96-8°, which, recrystd., yielded pure XXV, white needles, m. 97-8.5°, forming in Et2O with dry HCl in Et2O the HCl salt, m. 170-2°. XXV treated in CS2 with AlCl3 and BzCl on the water bath, the mixture hydrolyzed with ice, extracted with Et2O, the extract washed with aqueous NaOH, dried with CaCl2, evaporated, and the

residue

crystallized from petr. ether or EtOH yielded 25% 3,2,4,6-H2N(Me2CH)3C6HBz (XXVI), m. 128-30°. XXIV treated with excess EtI and Na2CO3 gave in the same manner 25% 2,4,6-(Me2CH)3C6H2NHET (XXVII), white needles, m. 80-2° (from absolute EtOH), yielding in dry Et2O with gaseous HCl the HCl salt, m. 262-3°. XXVII with BzCl and AlCl3 in CS2 yielded 69% 3,2,4,6-EtNH(Me2CH)3C6HBz (XXVIII), prisms, m. 124-6° (from petr. ether). VI (0.1 mol) in 322 cc. petr. ether treated dropwise with 0.1 mol Me2CO in 30 cc. petr. ether, and the mixture hydrolyzed after 0.5 h. and distilled gave 4 g. dimethylbornylcarbinol, b15 112-16°, nD18 1.4885, d18 0.9585, MRD 59.06,  $[\alpha]_D^{25}$  578 -13.4°,  $[\alpha]_D^{25}$  546 -16.6° (c 5%, EtOH). VI (0.117 mol) gave similarly with Et2CO 10.5 g. (40%) diethylbornylcarbinol, b25 152-6° nD17 1.4885,  $[\alpha]_D^{25}$  578 -13.8°,  $[\alpha]_D^{25}$  546 -16.2° (c 5, EtOH). VI (0.116 mol) treated with 14.3 g. Me3CCOCHMe2, b. 134-6°, and the mixture heated 17.5 h. and distilled gave 17.7 g. (60%) isopropyl-tert-butylbornylcarbinol, colorless liquid, b15 174-8°, nD16 1.5017,  $[\alpha]_D^{25}$  546 5.3° (c 5%, EtOH). VI (0.062 mol) in petr. ether heated 30 h. with an equivalent amount of (Me3C)2CO, b. 148-50° the mixture distilled, and the product, b15 190°, cooled and recrystd. from EtOH yielded 59% di-tert-butylbornylcarbinol, m. 70-1°,  $[\alpha]_D^{25}$  546 2.4° (c 1.66%, EtOH). A slight excess of PhAc in petr. ether treated dropwise with VI in petr. ether, and the mixture heated 6.25 h. and distilled twice gave 20% methylphenyl bornylcarbinol, b20 189-92°, nD14 1.5450, d14 1.022, MRD 81.5,  $[\alpha]_D^{25}$  578 13.4°,  $[\alpha]_D^{25}$  546 14.6° (c 4%, EtOH). Me3CBz and an equivalent amount of VI in petr. ether heated 10 min., the mixture carbonated immediately, hydrolyzed with H2O, let stand 20 h., and the organic layer distilled gave 38% tert-butylphenylbornylcarbinol (XXIX), colorless liquid with a bluish reflex, b25 210-14°, nD18 1.5388,  $[\alpha]_D^{25}$  578 10.8°,  $[\alpha]_D^{25}$  546 12.6°,  $[\alpha]_D^{25}$  436 22.4°; the petr. ether layer yielded an addnl. 8% impure XXIX, nD15 1.5270,  $[\alpha]_D^{25}$  578 22.2°,  $[\alpha]_D^{25}$  546 26°,  $[\alpha]_D^{25}$  436 47.4°. VI in petr. ether diluted with PhMe, the petr. ether distilled off, the residual mixture treated with 0.5 mol equivalent of XXVI, refluxed 7 h., hydrolyzed with H2O, the PhMe layer evaporated, and

the residue treated with aqueous HCl, Et<sub>2</sub>O, and acidified H<sub>2</sub>O, and purified through the free amine yielded 63% XXVI.HCl, m. 245-7°. VI in PhMe treated with 0.25 mol equivs. of XXVIII in PhMe, and the mixture heated 7 h. and worked up in the usual manner gave 83% recovered XXVIII, m. 80-2°; HCl salt, m. 262-3°. These results show that the reaction of VI with the sterically hindered ketones did not occur at the CO group but rather at another functional group. The reactions of the Li-organic compds. are compared with the known reactions of the corresponding Grignard derivs.

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TI Some derivatives of glucazidone

AU Maurer, Kurt; Schiedt, Bruno; Schroeter, H.

SO Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1937), 70B, 1857-61

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GI For diagram(s), see printed CA Issue.

AB The typical aromatic reagents attack glucazidone (I) (C. A. 29, 7986.5) in the pyridine nucleus, the 3-position apparently being the preferred point of attack in all cases. Only 1 substituent is introduced; if the conditions are made more drastic, the ring system is destroyed. Fuming H<sub>2</sub>SO<sub>4</sub> gives a yellow monosulfonic acid (II) which, unlike I, is easily soluble in water. Oxidative degradation with KMnO<sub>4</sub> splits off the SO<sub>3</sub>H group with formation of quinoxaline- $\alpha$ -carboxylic acid. While II is completely stable in dry form and gives a series of well crystallized salts, in solution it shows an interesting property. When it is allowed to stand in water in the dark, it remains unchanged, but in the light it soon becomes deep red and after some hrs. deposits a dark red, amorphous, alkali-insol. precipitate. The SO<sub>3</sub>H group cannot be replaced by HO, for with alkalis II is rapidly oxidized. Even in the absence of air, the product of alkali fusion rapidly decomps. When, however, II is nitrated the SO<sub>3</sub>H group is smoothly replaced by NO<sub>2</sub>, giving nitroglucazidone (III), which is also formed by nitration of I. III cannot be reduced to the amine because the whole ring system is very easily perhydrogenated. Catalytic hydrogenation gives an oily distillable octahydro derivative (IV), which, however, is unstable. I is readily halogenated. In the Br derivative (V) the Br is held quite firmly and cannot be replaced by heating with NH<sub>3</sub> or amines at high temps. and under pressure. V reacts with KOH in MeOH, but not homogeneously, and yields a halogen-free product (VI) of unsharp m. p. which is not identical with 3-hydroxyglucazidone. Chloroglucazidone (VII) is obtained from I with SO<sub>2</sub>Cl<sub>2</sub>. Nitration of V smoothly gives III. When Br reacts on II, there is apparently first 1,3-disubstitution immediately followed by saponification, giving a halogen- and S-free product with 2 HO groups,

assumed to be 1,3-dihydroxyglucazidone (VIII). VIII is easily soluble in alkalis and the solution eagerly absorbs O, becoming dark. The 2 HO groups can be etherified with CH<sub>2</sub>N<sub>2</sub> but not acylated. Attempts to benzoylate or acetylate VIII gave amorphous colored products which dyed wool. The reaction with CH<sub>2</sub>N<sub>2</sub> also does not proceed quite normally. Along with the etherification of the HO groups, the quinoid character of the ring system also manifests itself. Quinone, as is well known, adds 2 mols. CH<sub>2</sub>N<sub>2</sub> at the ring double bonds; in I, the addition occurs at only 1 double bond, as addition at the 1,11-position is sterically hindered. The product (IX) has the structure VIII, unlike I and the 3-HO derivative, reacts readily with PhNHNH<sub>2</sub>, giving a compound (X) with the composition, C<sub>18</sub>H<sub>14</sub>O<sub>2</sub>N<sub>4</sub>, of a normal phenylhydrazone. II (20 g. from 20 g. I slowly added with stirring and cooling to fuming H<sub>2</sub>SO<sub>4</sub> (20% SO<sub>3</sub>) and then heated 1.5 hrs. on the water bath), prisms with 1 H<sub>2</sub>O, m. 275°, previously darkening and decomposing K, Na, Ag salts. III (0.5 g. from 1 g. II slowly added to fuming HNO<sub>3</sub>), yellow, m. 215°. VIII (2.5 g. from 5 g. II in cold water treated dropwise with 4 atoms Br), faintly yellowish, m. 206°, difficultly soluble in water with acid reaction, soluble in concentrated acids with red color and reprecipitated by water, instantly reduces cold Fehling solution and KMnO<sub>4</sub> in acetone, gives with Ac<sub>2</sub>O in pyridine brown flocks decomposing above 280° and soluble in concentrated H<sub>2</sub>SO<sub>4</sub> with deep blue color;

dilution of the solution produces no flocculation but alkalinization results in a sharp color change. From 0.5 g. VIII with 50 cc. of a solution of  $\text{CH}_2\text{N}_2$  in ether there is obtained after 15 hrs. 0.35 g. of a yellow compound  $\text{C}_{14}\text{H}_{12}\text{O}_3\text{N}_4$ , m.  $186^\circ$ , soluble in alkalies, while 0.5 g. VIII suspended in 100 cc. ether and allowed to stand overnight with an ether solution of  $\text{CH}_2\text{N}_2$  from 5 g.  $\text{MeN}(\text{NO})\text{CONH}_2$  gives IX, m.  $99-100^\circ$ . X, yellow and red crystals, the yellow form becoming red on heating and m.  $202^\circ$ , soluble in concentrated acids with blue color, changing to violet and finally orange on dilution; alkalies give a yellow color. V (6.5 g. from 7.8 g. I and Br in benzene or  $\text{CHCl}_3$ ), yellow, m.  $172^\circ$ , sublimes at  $160^\circ$  in vacuo, forms with MeI at  $100^\circ$  a methiodide, red, m.  $194^\circ$  (decomposition), which gives a perchlorate, golden yellow, m.  $230^\circ$ , deflagrates vigorously when heated on a spatula. 9-Methyl-10-oxobromoglucosidone (1.3 g. from 2 g. V. MeI in water with alkaline  $\text{K}_3\text{Fe}(\text{CN})_6$ ), yellow, m.  $178^\circ$ , becomes discolored in the air.



10/591,778

=> log y

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

166.97

167.19

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE

TOTAL

ENTRY

SESSION

CA SUBSCRIBER PRICE

-40.80

-40.80

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